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MANUAL

OF

PRACTICAL ASSAYING,

INTENDED FOR THE USE

OF

METALLURGISTS, CAPTAINS OF MINES, AND ASSAYERS IN GENERAL.

WITH A COPIOUS TABLE, FOR THE PURPOSE OF ASCERTAINING IN ASSAYS OF GOLD AND SILVER THE PRECISE AMOUNT, IN OUNCES, PENNYWEIGHTS, AND GRAINS, OF NOBLE METAL CONTAINED IN ONE TON OF ORE FROM A GIVEN QUANTITY.

BY JOHN MITCHELL,

MEMBER OF THE CHEMICAL SOCIETY.

LONDON:

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PREFACE.

WHEN the rank our country holds among nations as regards her mining interest, is taken into consideration, it must be with all a matter of surprize that no work especially devoted to the elucidation of the processes to be employed in ascertaining the richness in metal of any sample of ore (that is, in other terms, its assay) has of late years appeared before the British Public. Indeed, the only work at present known in England is Berthier's "Traité des Essais par la Voie Sèche," which, for the mere purpose of inculcating the principles of assaying, has many disadvantages; not the least of which is its being written in a foreign tongue; and although a knowledge of French is now so very general, yet many parties are prevented from buying scientific works in that language on account of the difficulty of finding equivalents for the technicalities which must necessarily be employed. It is also a very

large work, and one containing much matter which the assayer does not heed, matter relating to the composition of wood and coal ashes, furnace products, &c., which are more especially adapted for the metallurgist.

These considerations, coupled with the paucity of any knowledge of assaying, excepting that confined to a very limited number of persons, induced the author of the following pages to turn a considerable amount of his attention to this subject, more especially as much difficulty was experienced in not having a suitable text-book for the use of his pupils. A portion of the following pages was drawn up as a Manual for such a purpose; but on consideration, it was thought the extension of such a work was so much needed, that it was determined to alter the original plan as far as was consistent with the complete carrying out the object in view, viz.: the production of a Manual embodying information in every branch of assaying, either by the wet or the dry processes.

The following is a sketch of the manner in which this is accomplished: the author having followed the excellent arrangement of Berthier as closely as possible, from whose work also much matter that suited these pages, and which it would have been useless to re-write, has been inserted. Firstly, the Mechanical and Chemical Operations of Assaying are treated in full, inclusive of a description of the apparatus required,

their mode of use, &c. Secondly, Furnaces, Fuel, and Crucibles, together with a description of the best Pyrometers and their applications. Thirdly, the Fluxes, their properties, preparation, use, &c. Fourthly, an Essay on the use of the Blow-pipe, and all its necessary appurtenances, as Fluxes, Supports, &c. Fifthly, the action of the Fluxes on some Mineral Substances. Sixthly, a method of discriminating many Minerals by means of the Blow-pipe, aided by a few tests by the humid method. Seventhly, the Humid Analysis of many Mineral Substances, their composition, locality, &c. (All the minerals mentioned in the three last heads comprehend such only as generally come under the notice of the assayer). Eighthly, the complete Assay of all the common Metals, in addition to which the Assay of Sulphur, Chromium, Arsenic, Heating Power of Fuel, &c. is fully discussed; and ninthly, and lastly, a copious table drawn up for the purpose of ascertaining in Assays of Gold and Silver the precise amount, in ounces, pennyweights, and grains, of Noble metal contained in a Ton of Ore from the assay of a given quantity. This table is the most complete and copious yet published.

Not only has it been endeavoured to collect all that is generally known on the subject of Assaying, but many new facts have been added, and such matter entered into, that the success of an assay is rendered

much more certain; and most assays are conducted more rapidly and with greater exactitude than heretofore.

It has also been endeavoured to introduce a new system, in which is pointed out the rationale of each process, with the chemical action taking place between the fluxes and the ores in course of assay, so that by paying a careful attention to the matters discussed, so much of the chemical nature of all ores that can come under the assayer's hand may be known, that the practice by "rule of thumb" (a rule on which very little dependance is to be placed, excepting after years of the most laborious practice, and a rule which cannot be imparted, excepting the pupil pursue the same unprofitable course) must, it is hoped, be speedily abandoned when, by knowing the chemical properties of the bodies operated on, the necessary fluxes and processes might be at once indicated, and with a certainty of perfect success.

Having premised thus much, the author must beg to express his thanks to his friend Mr. F. Field for the kind assistance he afforded him whilst experimenting on the various modes of assay described in the body of the work; and trusting that any little imperfections which may be detected will not be harshly criticised, but that it may be taken into consideration that the author has attempted to improve a branch of Mining

Knowledge to which unfortunately too little attention has been devoted, and to which, if he has added anything useful, he is indebted for the first principles of such knowledge to Berthier's "Traité des Essais," for which, to the talented writer of the above work he is under the most lasting obligation.

23, Hawley Road, Kentish Town,
London.

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THE MANUAL

OF

PRACTICAL ASSAYING.

CHAPTER I.

ON THE MECHANICAL AND CHEMICAL OPERATIONS OF ASSAYING.

In the process of assaying, the operations are of two kinds—*mechanical* and *chemical*. The ore before the operations of assay, in reality, has to be reduced to a fine state of division; this is effected by means of the anvil, hammer, mortar and pestle, sieve and decantation; or some of the means generally in use for the preparation of any fine powder; these constitute the mechanical operations.

The chemical operations come into play after the mechanical, and comprise calcination, roasting, reduction, fusion, sublimation, distillation, and cupellation. The apparatus employed in the chemical operations are generally crucibles of clay and metal, retorts, tubes, cupels, and furnaces.

The several operations will be described in the following pages, giving under each head such instructions, as, with ordinary care, will lead to accuracy in experiment.

MECHANICAL OPERATIONS.

Anvil and Hammer.—In general, the anvil and hammer are used for the purpose of breaking a small fragment from a mass of ore, or ascertaining whether a button of metal produced in an assay be malleable or brittle. The anvil is also exceedingly useful as a support for a crucible whilst breaking it to extract its metallic or other contents.

The most useful sized anvil weighs about 28 lbs., but one of 14 lbs. will suffice; its shape, that in ordinary use by the blacksmith. The hammer, or rather hammers, (for two are requisite), ought to have a flat square and a pick, or wedge end, the latter is useful for detaching fragments from a specimen of ore, and for breaking open crucibles; the former for flatting buttons of metal, or crushing ore on the anvil. The most convenient weight of the larger hammer is 4 lbs., and that of the smaller, 1 lb. The large hammer is also very useful for breaking coke sufficiently small for the use of the furnace, but both are used with the anvil according to the size of the mineral or crucible to be broken, or the button whose malleability is to be ascertained.

Very hard and stony minerals which have to be broken on the anvil (and all such ought) scatter many fragments to the certain loss of a portion of the substance, and the probable injury of the operator; this, however, can be prevented by wrapping the mineral in

a piece of stout brown paper, or, if necessary, in several pieces, the fracture can then be safely attempted. All minerals, unless very friable, ought to be thus treated before pulverization, otherwise, if they are broken in a mortar, it is nearly certain to be fractured, and, not only should the anvil be used for this purpose on the above account, but the operator will find his labour much abridged. The anvil can also be made very serviceable in re-pointing worn or burnt-out tongs, stirring irons, &c. It need scarcely be added, that the anvil must be placed on a firm block of wood, and, if possible, away from bottles and other frangible objects, otherwise accidents are liable to occur by the projection of fragments of crucibles, &c.

Pulverisation: the Mortar and Pestle.—Mortars are made of various materials, as cast-iron, bronze, porcelain, agate, &c.; but the assayer merely requires one of iron, and one of porcelain; and if he intend to use the blow-pipe in any of his operations, one of agate. The iron mortar ought to be of the capacity of a quart, the porcelain (Wedgewood ware) may be somewhat less, and the agate may be 3 inches in diameter; this size will be found the most useful, although one of 2 inches is considered by some parties to be as serviceable; the former size is, however, the better.

The ease with which a mortar may be used, depends much upon its form, and opinion is much divided upon this subject. Faraday* says of the pestle and mortar: "The pestle should be strong, and the size of its superior part such as may be sufficient to allow of its being grasped firmly in the hand, and below to permit a considerable

* Chemical Manipulation, p. 149.

grinding surface to come in contact with the mortar. Its diameter in the lower part may be about one-third or one-fourth of the upper diameter of the mortar, the curve at the bottom should be of shorter radius than the curve of the mortar, that it may not touch the mortar in more than one part, whilst, at the same time, the interval around may gradually increase, though not too rapidly, towards the upper part of the pestle."

The bottoms of all mortars ought to be of considerable thickness, in order to withstand the smart blows they will sometimes have to receive.

Iron mortars can best be cleansed by friction with a little fine sharp sand, if the ordinary process of washing be not sufficient to remove the adhering substance. Great care must be taken to perfectly dry mortars, especially those of iron, otherwise they will become rusted, and the rust so formed will contaminate the substances pulverized in them.

Berzelius recommends (and I have found it extremely serviceable) a mass of pumice stone for the cleansing of porcelain mortars. It is used with water as a pestle, and in course of time will be worn to the shape of the mortar, and then its action will be more speedy.

The iron mortar is principally of use in the reduction of the masses of mineral broken on the anvil to a state of coarse powder, in order to render the substance more readily capable of pulverization, strictly so called. In the use of the iron mortar, all friction with the pestle ought to be avoided, and the body within it must be struck repeatedly and lightly in a vertical direction, taking care to strike the larger pieces, so that all may be equally reduced. This process can be carried on until all are

about the size of fine sand; it can then be transferred to the porcelain mortar, where all blows must be carefully abstained from. The process is thus carried on: the pestle is to be pressed upon with a moderate force, and a circular motion given to it, taking care every now and then to lessen and enlarge the circles so as to pass over the whole bottom of the mortar, and ensure the pulverization of the mass of matter submitted to operation. The finer the state of division to which a mineral is reduced, the more accurate and expeditious will be its assay, so that no labour ought to be spared on this point. Pulverization is rendered much easier by operating on a small quantity at one time, and removing it very often from the sides and bottom of the mortar by means of a spatula. This quantity may be somewhat regulated by the hardness or friability of the substance operated on, the harder it is, the less must be taken, and *vice versâ*.

In the use of the iron mortar, fragments are occasionally projected; this may be prevented by covering the upper part of the mortar with a cloth. This applies also to the porcelain mortar, for the dust of some minerals has a disagreeable taste and smell; from this inconvenience, the operator may in some measure protect himself by means of the cloth. Indeed, in some cases, the ambient powder is highly poisonous, and the cloth is not a sufficient protection; under this circumstance, if the body to be pulverized is unacted upon by water, it must be moistened with that fluid, and the resulting mass dried for use.

Some minerals can be pulverized with greater ease if they be ignited and suddenly quenched in cold water;

in this list is the flint and many other silicious matters. In the pulverization of charcoal for assays, it will be found advantageous to ignite it, as hot charcoal is more readily pulverizable than cold.

Sifting : the Sieve.—The operation of sifting is had recourse to when a very fine powder is required, or when a powder whose parts must be equal is needed. A sieve is also very useful for the preparation of coke for the blast furnace. Sieves of various materials and degrees of fineness are necessary. The larger sieve for the coke may be made of stout iron wire, and have its meshes from 1 inch to 1½ inches square. One or two sieves may be provided, constructed of perforated zinc plate, these are very useful and economical; for very fine powder, a silk, lawn, or muslin sieve is essentially necessary. These latter may be conveniently and cheaply made by the operator. This is a desideratum, for the sharp angular particles of a mineral soon wear them out, yet they are preferable to the metallic sieves, inasmuch as they cannot contaminate anything they are used with.

The following simple contrivance constitutes the sieve I am in the habit of using; I take a common chip pill-box, of two ounce capacity, and strain over the end of it a piece of fine silk or muslin, (as the case may require), which is fastened round the box with string, in the same manner the paper cover is tied over an ordinary jelly-can—the excess of silk or muslin cut off near the string, and the bottom of the box cut out, the sieve is then finished. As they are so easily constructed, it is well to manufacture several of them, and keep one for copper, one for lead, tin, and so on. It is

convenient to close the aperture of the box with the lid when in use, for then none of the larger particles can be thrown over the sides and injure the sieved product.

Where the powder to be sifted is deleterious or offensive to the operator, a sieve termed a *drum*, or *box sieve*, may be substituted. It consists essentially of a box in the shape of a drum, covered tightly at both ends with stout leather; the box is divided into two equal compartments by a sieve, which may be separated entirely from either end of the box: the sieve has a ledge which fits the lower compartment, and one fitting the upper, so that it is perfectly close, and when in use, no unpleasant effect can take place. The method of using this sieve is, however, rather peculiar, and requires some practice to fully develop its powers. One side of the under edge must be held by one or both hands, according to its size, whilst the other side rests on a table or bench; a semicircular oscillating motion must now be communicated to it by moving the hands up and down, at the same time they are being alternately brought into approximation with the sides of the operator.

The use of the ordinary sieve is so well known that it does not require description.

The sieve is extremely serviceable in the separation of some ores from their gangues, especially if the latter be very hard and stony. This point must be particularly noted, as it is the cause of much variance between the results of different assayers—for instance, part of the same sample of ore might be sent to two assayers, and the produce made by one might be 8½ per cent., and that made by the other, 9, or 9½, or in some cases even more. This discrepancy most probably arises from the

cause just mentioned; in the one case, part of the hard gangue might be rejected, and so render the residue richer; and in the other the whole might be pulverized, and the produce would be of course less, and nearer the actual amount contained in the sample submitted to assay.

A knowledge of this fact is also very useful, but in another point of view: suppose it were wished to separate, in a speedy manner, any friable mineral, such as galena or copper pyrites, as perfectly as possible by mechanical means; we should employ the sieve. The method of operating is as follows: place a small quantity of the mineral in an iron mortar, and strike repeatedly, slight vertical blows. When it is tolerably reduced, sift it; that which passes through is nearly pure mineral, with only a small quantity of matrix, repeat the pounding and sifting processes, until after a few alternations, that which remains on the sieve is nearly pure gangue.

Decantation.—This process can only be employed for those bodies which are not acted upon by water, and is effected thus: the substance operated on is reduced to the finest possible state of division by means of the foregone processes; it is then mixed with a quantity of water in a glass or other vessel; after a few moments' repose, the supernatant liquid, containing in suspension the finer particles of the pulverized substance, is poured off, and the grosser parts, which have fallen to the bottom of the vessel, are repulverized, and decantation again made use of. By alternating these processes, the finest possible powder may be obtained in a ready manner; it is seldom, however, that a substance is required in such a minute state of division, as produced by this process,

for assaying by the dry way; in the humid method it is occasionally very useful. Decantation is not only employed for the above purpose, but as a ready means of separating a liquid from a precipitate in an assay by the humid method, or in washing a precipitate with a large quantity of water, in order to free it from any adhering impurity which is soluble in that fluid. In certain cases, where the precipitate to be washed is light, the least disturbance of the vessel containing it occasions its distribution in the liquid, and the consequent loss of a portion in decantation; this can be avoided by the employment of the syphon. The operation is then thus conducted. The syphon is filled with water, and the shorter end placed in the liquid, whose transvasion is to be effected, the forefinger of the right hand being, during this time, applied to the longer end of the instrument; it is now to be removed, and the water will flow out until it be level with the immersed end of the syphon. Fresh water can then be added to the precipitate, and the operation of decantation by the syphon carried on as long as requisite.

CHEMICAL OPERATIONS.

Calcination.—This operation has for its object the separation of any species of volatile matter, as water, bitumen, arsenic, &c., from a mineral substance; the atmosphere being excluded, or the ignition of any substance, as quartz, &c., previous to its quenching in water. The hydrates of iron, zinc, and all minerals whose matrices are argillaceous, are calcined for the purpose of expelling combined water. Carbonates of lead, iron,

zinc, and lime, are calcined to expel carbonic acid and water; cobalt, nickel, &c., to separate arsenic and sulphur; coal, and the iron ore found in the vicinity of collieries, to expel bituminous matter.

Vessels termed crucibles are used in calcination, and are made of various materials, as clay, platinum, silver, and iron. Silver cannot be employed where a heat greater than dull redness is requisite. Crucibles of platinum are the most useful; next to them, those of clay. All these crucibles must be furnished with covers.

When the operation is finished, the crucible must be taken from the fire, and allowed to cool gradually; when completely cold, the cover may be removed and the contents taken out with a spatula; if any adhere, a small brush will be found very useful for its removal. The difference in weight, before and after its calcination, equals the quantity of volatile matter.

In case the substance to be calcined is fusible, the crucible and contents must be weighed before ignition, and the loss of weight is equal to the quantity of volatile matter expelled. If the ignited substance be soluble in water, it can be removed from the crucible by that menstruum, (heat may be employed if required), if not, an acid may be used.

It may be as well to state in this part, as we have mentioned the use of crucibles made of platinum, that certain bodies cannot be ignited in them; and the best instructions given for their use are those of Berzelius. "It is improper to ignite, in platinum vessels, the caustic alkalies, or the nitrates of any alkaline base, such as lime, baryta, or strontia, because the affinity of the alkali for the oxide of platinum causes a very consider-

able oxidation of the metal, and after the saline matter is removed, the surface of the metal is found to be honey-combed. The alkaline sulphurets, or the alkaline sulphates mixed with charcoal, are inadmissible, because the sulphurets, so formed, attack platinum more energetically than even the caustic alkalies. Again, metals whose fusing point is lower than that of platinum, because an alloy would be formed. Gold, silver, and copper may be heated to a dull redness, in platinum vessels, without danger; but fused lead cannot come in contact with platinum without destroying it. A drop of fused lead, tin, zinc, or bismuth, placed on red hot platinum, always produces a hole. Neither can phosphorus or phosphoric acid, mixed with charcoal, be ignited in vessels of platinum, because a phosphuret of platinum is produced, which is an exceedingly brittle compound.

"In analyses by the humid method, nitro-hydrochloric acid (*aqua regia*), even when very dilute, must not be allowed to come into contact with platinum. It is a general rule, that liquids containing either chlorine, iodine, or bromine, must not be boiled in platinum capsules."

The best method of cleaning a platinum capsule or crucible, when it has become scoriaceous, is to smear it over with a paste made of borax and carbonate of soda, and then heat it to redness; after it has cooled, place it in boiling water until all the saline matter is dissolved, when the vessel will be generally found bright and clean; if not, the operation must be repeated.

Roasting.—In this operation, arsenic, sulphur, selenium, carbon, and antimony, are separated from certain

metals with which they were combined. Roasting differs from calcination in this particular, the latter is carried on in close vessels independent of the atmosphere, the former in open vessels by the aid of the atmosphere. It is thus we are enabled to separate the bodies just mentioned by this process; for the oxygen of the air, in combining with them, forms a volatile substance, which the heat employed, expels.

In roasting, in the ordinary acceptation of the term, the body must *not* be fused, but kept in a pulverulent state; but there are roastings where the substance *is* fused, as in cupellation and scorification. The process of roasting is performed in various ways. In one, a small flat vessel, similar to a saucer, is employed (made of the same material as the earthen crucibles), which is placed in a muffle, or on the bars of a calcining furnace. The body to be roasted must be finely pulverized, and when the roasting vessel is at a dull red heat, may be introduced, and constantly stirred with an iron rod until no fumes are given off. It may also be performed in a crucible, in a common furnace, taking care to place the crucible a little inclined on one side, and stirring the material with a rod, as before.

The heat, in this operation, must be very nicely regulated for some time; at first it ought only to be the dullest red, and the substance must be assiduously stirred, in order to present the largest possible surface to the action of the atmosphere, and prevent fusion; for some assays, when roasting, will fuse very readily at a low temperature, unless the surface be continually renewed. If the assay at all agglutinates, it must be taken from the fire, and recommenced,

if the substance be plentiful; but if not, the fused mass must be carefully removed from the crucible, pulverized, and the roasting recommenced. In this case, however, the operation is always rendered very tedious, so that much care ought to be taken at the commencement of the roasting. After the assay has continued at a dull red heat for some time, and shows no inclination to agglutinate, the heat may be slightly increased; at the same time, the stirring must be diligently pursued. After the heat has arrived at full redness, there is little fear of fusion; and as the operation proceeds more rapidly towards the end, at a high temperature than a low one, it is well at this point to increase the heat to a yellowish red, and lastly, in certain cases, to nearly a white heat. If the stirring of the assay has been constant during the various gradations of heat, the roasting at this point will be accomplished, and the remaining operations of the assay may be proceeded with.

There are certain other precautions to be taken in roasting some minerals, which will be pointed out under the assay of their respective metals. It may be as well to mention here, that platinum capsules are useful in certain roasting operations; the sulphurets of copper, iron, and molybdenum, are most conveniently oxidized in this kind of vessel without fear of injury to it, provided that fusion of the roasting substance be carefully avoided. Platinum vessels must also be used in ascertaining the amount of ash in coal, where the experiment is conducted so as to afford exact results.

Reduction.—The process of reduction consists in removing oxygen from any body containing it, by means either of carbonaceous matter or hydrogen, or a body

containing both of these elements. Carbon, however, is the reducing substance generally made use of.

In the operation of reduction by the aid of carbonaceous matters, two methods are employed; in the one, charcoal, coal, or any carbonaceous, or hydrocarbonaceous body is mixed with the substance to be reduced, and the whole placed in the fire; in the other, the process of cementation is employed, as in the manufacture of steel. This process is conducted by placing the oxide in a crucible lined with charcoal, and covering it closely during the time it is in the furnace; the reduction proceeds gradually from the outside of the oxide, in contact with the charcoal, to the centre of the mass. The time requisite for this operation depends on three circumstances, viz.: the nature of the oxide, the degree of temperature, and the mass acted upon.

Some oxides treated in this manner are reduced very readily; others, again, take a considerable time; while certain of them do not appear to be acted upon beyond the outermost layer. Of the first class, is oxide of nickel; of the second, oxide of manganese; and of the third and last, the oxides of chromium and cerium.

Each of these classes of reduction has its advantage. The former, or reduction by mixture with carbonaceous matter, takes place very quickly and completely, but the metallic residue is mixed with charcoal; the residue by the latter process is comparatively pure, but it is not generally preferred, on account of the time and high temperature necessary.

Fusion.—This operation is sufficiently simple, and is employed in all assays by the dry way, in order to

obtain in conjunction with reduction, a button (as it is termed) of the metal whose assay is in progress. It is also a necessary step in the granulation of metals, and the preparation of certain fluxes, alloys, &c.

Sublimation.—This operation is a kind of distillation, in which the product is obtained under the solid form. The apparatus generally employed for this purpose are tubes, flasks, capsules, and crucibles. Flasks, (those in which Florence oil is imported), are exceedingly useful; they must be sunk in a sand-bath, and the sublimed substance received directly into another flask, or by passing through an intermediate tube. Sometimes, however, it is difficult to entirely remove the sublimed substance; and in order to avoid this inconvenience, Dr. Ure has proposed the following very excellent subliming apparatus. It consists of two metallic or other vessels, one of which is flatter and larger than the other. The substance to be sublimed is placed in the smaller vessel, and its opening is covered by the larger, filled with cold water, which may be replaced from time to time as it becomes hot. The sublimed substance is found on the lower part of the upper vessel. A large platinum crucible, filled with cold water, and placed on the top of a smaller one, answers the purpose of the before-mentioned apparatus very well.

Distillation.—There exist two distinct classes of this operation; in the one, liquids are submitted to experiment; in the other, solid bodies, such as wood, coal, &c., in order generally to ascertain the amount of gas, or other volatile matter given off in the course of an experiment from a certain quantity.

In liquid distillations, glass vessels, termed retorts, are used. The best for general use are furnished with a stopper at the upper part of the body, through which the liquid is introduced; the neck of the retort is then placed in that of a receiver, which must be kept cold by means of a stream of water; heat is then applied to the retort, and as much of the liquid as is desired is distilled over into the receiver. It is advisable to fill the retort no more than two thirds full, and to apply the heat, at first, very gently, otherwise there is a fear of breaking the vessel.

In the distillation of dry bodies, earthenware retorts are sometimes employed, but in general cases, I find a tube of wrought iron, plugged at one end, to be the most convenient form of apparatus. It is placed, with the substance containing it, in the fire, and a small tube, either of glass or pewter, is fixed by means of a cork to the open end of the large tube. The gas given off during the operation is to be collected at the pneumatic trough. It will be necessary here to describe the pneumatic trough and jars, together with all the necessary calculations for temperature, pressure, and moisture, requisite in experimenting with gaseous bodies.

The pneumatic trough is a vessel of either a circular or square form, (the latter is most convenient), furnished with a shelf at the distance of about three inches from its upper part; this shelf, according to its size, is perforated with one, two, or more holes, each of which is furnished with a small funnel-shaped opening on the inferior part; this opening is for the purpose of receiving the mouth of a tube delivering gas The lower part

of the trough ought to be furnished with a tap, for the purpose of drawing off the water when it is soiled. The gas jars are made of glass, (the most convenient form is cylindrical), and graduated to cubic inches and parts; each of the jars may hold from 50 to 100 cubic inches, or more, according to the quantity of gas expected to be furnished during each experiment.

To use the trough, proceed as follows:—Fill it with water to about two inches above the shelf; then fill one of the jars with water and place it in an inverted position on the shelf over one of the holes with the funnel-shaped opening, into which introduce the gas-delivering tube. As soon as the gas passes off by the aid of heat from the coal, or other body in the iron tube, it will pass into the jar and displace the water. As soon as the jar is full it must be replaced by another, and so on until no more gas passes over. The quantity produced in the experiment is then ascertained; it is not, however, the true quantity, as most likely it has been expanded to a larger volume by the heat employed in its production, or has combined with a quantity of aqueous vapour from the water with which it was in contact; and lastly, the barometer might not have been at the height of 30 inches from some change in the state of the atmosphere; if it were less than 30 inches, the gas would appear greater in quantity; if more than 30 inches, it would appear less in quantity than it really was. The following is the method of making the calculations necessary in reducing the gas to its true volume.

Correction for Temperature.—It has been ascertained by Dalton and Gay Lussac, that 100 parts

of air, or any other gas, at 32° of Fahrenheit, when heated to 212°, expand to 137.5 parts, the increase being $\frac{37.5}{100}$th, or .375 of the original bulk. If this be divided by 180, the number of degrees between 32° and 212°, it will be found that air expands $\frac{1}{480}$th for each degree of Fahrenheit, and we can, from this datum, determine the expansion or contraction any gas would undergo for any given number of degrees of temperature. But supposing it be required to know what volume 100 cubic inches of gas at 80° would occupy at 60°, the standard temperature, it must be kept in view that it is not $\frac{1}{480}$th part per degree of the volume at 80°, but of the volume at 32°, which is to be deducted; 480 parts of air at 32°, become 481 at 33°, and 482 at 34°, and so on, so that at 60° they have increased to 508 parts, and at 80° to 528, so that we have a proportion between the bulks at 60° and at 80°, from whence the question may be determined: for

Volume at 80°.		Vol. at 60°.		Cubic inches.		Cubic inches.
480+48	:	480+20	::	100	:	96.212

or the reverse, supposing it were wished to ascertain the real volume at 60° of 100 cubic inches of gas observed at 40°.

Volume at 40°.		Vol. at 60°.		Cubic inches.		Cubic inches.
480+8	:	480+28	::	100	:	104.098.

Correction for Pressure.—As before stated, the standard pressure is 30 inches of mercury, and the law must be kept in mind that the bulk of a body of gas is inversely proportionate to the weight and directly proportionate to the pressure, so that if we had 100 cubic inches of air when the barometer was 29 inches, it would

be as 30 : 29 :: 100 : 96.6, or if the barometer were at 31 inches when the 100 cubic inches were measured, it would be as 30 : 31 :: 100 : 103.33, so that the rule is as the mean pressure is to the observed pressure, so is the observed volume to the true volume. The correction for temperature or pressure may be made indiscriminately, the result being the same in either case.

Correction for Moisture.—This correction must be made after the two previous. As before-mentioned, the elastic force of the aqueous vapour causes the gas with which it may be mixed to expand, and by reference to tables founded on calculations upon the force of steam at different temperatures, the amount of the correction may be easily ascertained.* Thus, for 100 cubic inches of a gas saturated with vapour, properly corrected to the temperature of 60°, and 30 inches pressure, we wish to know the equivalent bulk of the dry gas. The observed volume is partly due to the expansion occasioned by the vapour, and this portion will be in proportion to the whole, as the elasticity of the vapour is to the total elasticity: therefore,

Elasticity of air.		Elasticity of vapour.		Cubic inches.		Cubic inches.
30.000	:	0.560	::	100	:	1.86

The volume of the dry gas is therefore:

$$100 - 1.86 = 98.14 \text{ cubic inches.}$$

Cupellation.—This operation will be described under the head, Silver Assay.

* Daniell's Introduction to Chemical Philosophy.

CHAPTER II.

FURNACES, FUEL, CRUCIBLES, &c.

FURNACES are of two distinct kinds, viz.: *blast* and *wind*. In the former, the fire is urged by means of bellows; and in the latter, by a chimney, or common draught. We shall commence with the latter, as they are in most common use. They are of various kinds according to the purpose for which they are required. The three principal kinds are those of fusion, calcination and cupellation. Coal, coke, or charcoal are the fuels employed, and the merits of each will be particularly discussed. Blast furnaces are only employed for the purpose of fusion, although their forms are various: charcoal and coke are the fuels in use.

All furnaces consist of certain essential parts, viz.: 1st. the ash pit, or part destined to contain the refuse of the combustible employed; 2nd. the bars on which the fuel rests; these are sometimes made moveable, or fixed to a frame, the former arrangement is more convenient, as it allows clinkers and other refuse matters to be readily removed. 3rd. The crucible, or body of the furnace in which the heat is produced; and lastly, in wind furnaces, the chimney by which the heated air and gaseous products of combustion are carried off.

Wind Furnaces: Calcining Furnace.—Calcining furnaces are small and shallow, because a high temperature is not required. They may be made square or circular. The former are most readily constructed, and the fuel they contain can be easily stirred without fear of overturning the contained crucibles. Where many crucibles are to be heated at once, they are preferable to the circular, but the latter give the greatest degree of heat with the least possible consumption of fuel, and are to be preferred on that account, where one crucible only is to be ignited.

The *crucible*, or *body* of the furnace is best made with good bricks, lined with Welsh lump, fire-bricks, or a mixture of Stourbridge clay and sand. It is also desirable that a plate of iron with a ledge be placed over the upper part of the furnace to protect the brickwork from blows with crucible, tongs, &c., and to keep it in its place when disturbed by sudden alternations of temperature. The bars of the furnace may be either one single piece, or made up of several bars of iron fastened to a frame. They ought to be as far as may be from each other, and not be too large. They must be large enough, however, not to bend under the weight of the fuel and crucibles when they become hot, and they must not be so far removed from each other as to allow the coke or charcoal to fall through readily. Lastly, the more readily the air can find access to the centre of the fuel, the higher will be the temperature produced in the furnace, and very simple assays occasionally fail, only because the bars are either too large or too close together.

The *ash-pit*, as before stated, is an open space under

the bars, and serves as a receptacle for ashes, clinkers, &c., produced during the time the furnace is in use. It ought to have the same area as the crucible, and completely open, so that the air may have free access; it is well, however, for the sake of economy, to furnish this opening with a hinged door, having a register plate fixed in it, so that the draught may be reduced, or entirely shut off, in order that the fire may be extinguished and fuel saved, which otherwise would be burnt in sheer waste.

Chimney.—Calcining furnaces generally have no fixed chimney, but are covered with a moveable one when a greater degree of heat is required. This chimney is made of strong plate iron, furnished with a wooden handle. The lower part is provided with a door, by means of which the interior of the furnace may be examined without disturbing the whole arrangement of the chimney, and consequent cooling of the contents of the furnace.

If, during the course of any experiment, noxious or inconvenient vapours are expected to be given off, the furnace must be so arranged that they are introduced into a flue by fastening a piece of iron plate pipe furnished with an elbow joint on to the moveable chimney before spoken of.

Evaporating Furnaces.—The furnaces just described answer exceedingly well for heating small flasks, evaporating basins, &c., when furnished with a tripod stand or sand-bath. The latter is necessary, as many assays by the dry way are preceded or followed by certain operations by the humid method.

The Hood.—In order to prevent certain vapours from

fires, evaporating basins, &c., from entering into the laboratory, a large metal covering, termed a hood, is employed, which hood terminates in a chimney, having a good draught. They are best made of sheet zinc, plate iron, or better still, of galvanized iron, as that is cheaper than zinc and quite as serviceable; it has also the advantage of not being combustible.

Fusion Furnace: Wind Furnace.—The wind furnace, properly so called, is a furnace provided with a chimney, and which is capable of producing a very high temperature.

Wind furnaces are generally square, but if more than four crucibles are to be heated at one time, they may be made rectangular, the chimney being placed at one of the long sides. However, when the furnace is required to hold but one pot, it may be made circular.

The body of the furnace ought to be made of good bricks, solidly cemented with clay, and bound by strong iron bands. The bricks must be very refractory and capable of sustaining changes of temperature without cracking. They are ordinarily made with the clay used in the manufacture of crucibles. In some cases, bricks are not used for the lining of this kind of furnace; for instance, a mould of wood is placed in the centre, and the open space between the surface of that and the outer brickwork is filled with a paste of very refractory clay, each layer being well beaten down. When the space is filled, the case is withdrawn, and the crust of clay dried with much precaution, every crack that may be caused by unequal dessication, being filled up as fast as formed. This method of manufacture is very appli-

cable to circular furnaces. In every case, however, it is necessary to border the edge with a band of iron, to prevent injuries from tongs or pots.

The Ash-pit.—On the one hand it is well to have the power of cutting off access of air into the body of the furnace by the lower part, either to put out the fire entirely, or to deaden it whilst putting in a pot, and on the other, to attain the maximum of temperature, we must have the means of allowing the air to pass with the greatest possible facility into the furnace. In order to do this, it is necessary to furnish the ash-pit with doors, or valves, whereby the quantity admitted may be regulated as described.

The *bars* are made in one piece, or are made up of moveable pieces of metal, which latter arrangement is the most convenient. Wherever a wind furnace is in use, the superior opening is closed by a cover made of refractory bricks, encircled with iron.

The *chimney* is a very essential part of a wind furnace; it is on its height and size that the draught depends; and, in consequence, the degree of heat produced within the furnace. In general, the higher and larger the chimney is, the stronger is the draught, so that by giving it a great elevation, exceedingly high temperatures may be obtained; but there is a limit which it is useless to pass in a furnace destined for operations by the dry way, and besides this, the building a very high chimney presents many difficulties and much expence, so that in laboratory operations, where a very strong current of air is required, recourse is had to a pair of double bellows. A temperature can be produced in a wind furnace sufficiently strong to

soften the most refractory crucibles by means of a chimney from thirty-six to forty feet high. They are generally made square or rectangular, and have interiorly the same dimensions as the crucible of the furnace. About two feet above the upper part of the furnace, these chimneys are furnished with a register, or damper, by means of which the current of air may be regulated, or entirely stopped at will. The damper is a plate of iron sliding into a small opening across the chimney.

Blast Furnaces.—In this species of furnace, the air necessary to keep up the combustion is forced through the fuel by means of a blowing apparatus, instead of being introduced by the draught of a chimney, as in the wind furnace.

The most convenient apparatus for forcing air into a furnace is a double bellows; a fan may be used, but, in the small way, is not so powerful.

The quantity of air passing into a furnace varies with the length of the assay, and ought to increase gradually, the stream at first being small, and as the temperature becomes higher, the bellows ought to be worked with more force.

The following is the description of a most excellent blast furnace which has been in use for some years in the laboratory of the Royal Institution. I have in my own laboratory one of these furnaces, and the temperature produced by it is extraordinary, considering the small amount of time and fuel employed. It is sufficiently powerful to melt pure iron in a crucible in ten to fifteen minutes, the fire having been previously lighted. It will effect the fusion of

rhodium, and even pieces of pure platinum have sunk together into one button in a crucible subjected to its heat.* All kinds of crucibles, including the Cornish and Hessian, soften, fuse, and become frothy in it; and it is the want of vessels which has hitherto put a limit to its application. The exterior consists of a blue pot, (black-lead pot), eighteen inches in height, and thirteen inches in external diameter at the top; a small blue pot of seven and a half inches external diameter at the top, had the lower part cut off so as to leave an aperture of five inches. This, when put into the larger pot, rested upon its lower external edge, the tops of the two being level. The interval between them, which gradually increased from the lower to the upper part, was filled with pulverized glass-blowers' pots, to which enough water had been added to moisten the powder, which was pressed down by sticks, so as to make the whole a compact mass. A round grate was then dropped into the furnace, of such a size that it rested about an inch above the lower edge of the inner pot, the space beneath it, therefore, constituted the air chamber, and the part above, the body of the furnace. The former was $7\frac{1}{2}$ inches from the grate to the bottom, and the latter $7\frac{1}{2}$ inches from the grate to the top. Finally, a longitudinal hole, conical in form, and $1\frac{1}{2}$ inches in diameter in the exterior, was cut through the outer pot, forming an opening into the air chamber at the lower part; its use being to receive the nozzle of the bellows by which the draught was thrown in.

The Muffle, or Cupel Furnace is a species of rever-

* Faraday.

beratory furnace, in the centre of which is placed a small semi-cylindrical oven which is termed the muffle.

This muffle being completely surrounded by ignited fuel acquires a very high temperature, and in its interior, all operations requiring the presence of air, and which may not be carried on in contact with carbonaceous matters may be performed, such as roastings, scorifications and cupellations.

When from ten to twenty cupellations have to be effected at one time, large brick furnaces are employed; and, in consequence, much fuel is consumed to waste, where only a few cupellations are required. This has occasioned many persons to endeavour to form small furnaces, where one or two cupellations may be carried on with the smallest possible quantity of fuel. MM. Aufrye and d'Arcet have contrived a furnace which is capable of fulfilling all these conditions.

The furnace is elliptical and about 7 inches wide and 18 high; its ash-pit has but one circular opening, and its height is such, that when the furnace is placed upon it, and the whole upon a table, the assayer can, when seated, readily observe the course of the assay within the muffle. The hearth has five openings, in one of which the muffle is placed; in another, a brick to support it; a third for the purpose of introducing a poker to stir the ashes, and make them fall through the grate-holes; this can be closed with a small earthen plug; and lastly, two round holes, placed in its largest diameter, to facilitate the introduction of air, either by draught or a pair of bellows, as the case may require. The support for the fuel is generally a plate of earthenware, pierced with holes, and bound round with iron

wire to keep it together in case it cracks by changes of temperature; but it is better to use an iron grating.

The dome of the furnace has a circular opening, which can be closed by a plug of earthenware; this opening serves for the introduction of the fuel. A chimney is necessary to increase the draught; it is made of sheet iron, and may be from 1½ to 2 feet in height, and ought to fit the upper part of the dome very exactly. At its base there is a small gallery, also of sheet iron, in which is intended to be placed the new cupels, so that they may be strongly heated before introduction to the muffle. This saves many of them from fracture.

MM. Aufryé and d'Arcet have estimated the quantity of charcoal necessary to heat this furnace. The following are comparative experiments.

No.	Silver employed. grains.	Lead employed. grains.	Time of assay. minutes.	Standard. thousandths.	Charcoal used. grains.
1	1	4	12	947	173
2	1	4	11	950	86
3	1	4	13	949	93
4	1	4	10	949	60

Coke or charcoal may be used in this furnace, but the fire must be lighted by means of charcoal alone, because coke is very difficult to inflame in a cold furnace. When it is red hot, it may be fed with coke alone, or better still, a mixture of coke and charcoal.

AUXILIARY APPARATUS.

Ordinary assay furnaces require very few instruments; they are: firstly, pokers, or stirring rods, made of stout bar-iron; these may be straight, as for stirring

the fuel from the top of the furnace, or curved for clearing the bars from clinkers or ashes; secondly, straight and curved tongs, of different sizes and forms, for placing crucibles, &c., in the fire; thirdly, sheet iron shovels, for the purpose of introducing coke and other fuel into the furnace; they are best provided with holes of small diameter, so that coke and charcoal may be separated from dust by sifting before it is burnt; and lastly, a wooden mask, furnished with glass openings, through which the state of the fire, &c., may be ascertained without injury to the face. The glass is best of a deep neutral tint; a pair of neutral tint spectacles are very serviceable, when the eyes alone are wished to be protected.

In cupel furnaces, pokers or stirring rods, both curved and straight, are required, as also a mask or spectacles. The tongs are varied in form; thus we have the ordinary straight tongs for the purpose of holding fuel, hot doors, &c., then light tongs, whose nose is slightly curved, for introducing and withdrawing the cupels; also very light elastic pincers to place the assay buttons in the cupel; and lastly, small iron spoons, for the introduction of granulated lead.

I will now describe the furnace I am in the habit of using. It may very aptly be termed the "universal furnace," as it is capable of performing all that is required of any furnace in an assay. It is to be much recommended for its durability and cheapness, and lastly, for its small size, compared with the heat it can produce. It is made, I believe, only by Mr. Newman of Regent Street, and is constructed externally of sheet iron, very stout, and is lined with fire brick, not cemented

together, but ground and keyed, as an arch, so that it can never fall out until it is completely useless. It is furnished with five doors, one in the ash-pit, and four in the body of the furnace; two in the front, one above the other, and two opposite each other, at the sides. The cover serves as a sand-bath, and when that is taken off, there is a series of cast iron rings, fitting the top of the furnace, for the purpose of placing basins either for the purpose of evaporation, calcination, or roasting. The two opposite holes serve for the introduction of a tube in operations where it is requisite to pass a gas over any body, at a red heat. In the lower hole, in front, I place my muffle for roastings and cupellations, and introduce fuel and crucibles by the upper one; it also serves as an opening through which the state of the furnace can be seen, or the progress of any assay observed.

Iron, manganese, nickel, and cobalt, can be fused in this furnace, when it has a flue of about thirty feet in height attached to it; and by closing the ash-pit door, the dullest red heat, for gentle roastings, can be obtained. Its height is about 2¼ feet, and diameter 1 foot; internal diameter, 8 inches, and depth of fire-place 1¼ ft.

The use of Furnaces.—Care must be taken in placing the crucibles in the fire. They must stand solidly, and be at equal distances from the sides and bottom of the furnace, so as to receive a like share of heat, and they must be completely surrounded with the fuel. The fire must be got up gradually, so as to prevent the sides of the furnace and the crucibles within from cracking from the sudden increase of heat. After the furnace has arrived at a full red heat, give more air, and in from about twenty

minutes to one hour, the assay will be finished. During the time the furnace is in full action, the cover must be frequently removed to add more fuel, if any open spaces occur round the crucibles, also to press the fuel close to the pots. When the pots are taken out, they may be placed in a sand-bath, and allowed to cool gradually, before they are broken, to examine the contents.

In commencing a second assay immediately in the same furnace, certain precautions must be taken to ensure success. In the first place, all ash and clinker must be removed from the grate, by means of a crooked poker; secondly, the fuel must be pressed down firmly; and lastly, a layer of fresh combustible must be placed on the fire, and before that is ignited, the crucibles must be arranged upon it, and the spaces about them be filled with coke or charcoal, as the case may be, and the assay proceeded with as before.

In executing many assays, one after the other, a great saving of fuel is made, for the furnace is not allowed time to cool.

FUEL.

Assay furnaces are heated with coal, coke, and charcoal, and sometimes with a mixture of the two latter; coal is very seldom employed, and therefore will not be much spoken of; coke is the principal combustible used in assaying. Calcining furnaces ought to be heated with charcoal alone, because coke employed in small quantities lights and burns with too much difficulty. All fuels contain certain fixed matters, which remain after combustion, and which constitute the ash. These ashes fuse or agglutinate together, and when a certain quan-

tity is formed, if it be not removed, the fire will decrease in intensity, and finally die out. As all combustibles do not contain the same amount of ash, we must choose amongst them, and those containing the least are to be preferred; in the first place, because weight for weight, they contain more available matter; and secondly, because they can be used in a furnace a longer time without the formation of so much clinker. The composition of the ash merits much attention.

Charcoal contains from 3 to 4 per cent. of ash in general, the components of which are lime and potash, as carbonates. It is true, certain other matters are present, as phosphoric acid, oxide of iron, manganese, &c., but in very minute proportions. It is not fusible *per se*, and if it do not meet with any substance capable of combining with it, it passes through the bars as a white powder, but when the potash predominates, it exercises a very corrosive action on the bricks with which the furnace is lined, as also on crucibles, lutes, &c., by the formation of a fusible silicate of potash, which in course of time runs down the sides of the furnace, and chokes the bars. Whenever the ash is in very small proportion to the charcoal, its fusion is rather useful than otherwise, because a species of varnish is found, which penetrating into the substance of the bricks and lutes, gives them solidity by binding them together with a cement, forming part of their substance.

The proportion of ash which coke contains is very variable; that of commerce contains from 8 to 10 per cent., while some samples of coke, made from very pure coal, give but 2 to 3 per cent., so that this fuel ought to

be carefully chosen. The nature of this ash is different to that of charcoal; it consists principally of oxide of iron and clay. The former is produced from the pyrites coal generally contains. The clay is similar to the carbonaceous schists, not very fusible by itself, but nevertheless capable of softening. When pure, it forms a slag, which attacks neither the bricks or crucibles. This happens very rarely; it is more often that oxide of iron predominates, which by contact with the carbonaceous matter becomes reduced to the state of protoxide, and then not only becomes very fusible, but exercises on all argillaceous matters a very corrosive action, so that crucibles are very seriously injured, and the sides of the furnace require frequent repairs.

Weight for weight, coke and charcoal give out nearly the same quantity of heat; but in equal bulks, the former developes much more heat, because its density is greater; so from this difference in the calorific power of coke and charcoal, it results, that in the same furnace, the former produces a greater degree of heat than the latter; and it has been proved, that at high temperatures, the difference is nearly 10 per cent. In order to account for this, we must consider, firstly, that in a given space the quantity of heat produced in a certain time, and, in consequence, the temperature depends on the weight of fuel burnt, and increases with its weight; secondly, that combustion taking place but at the surface of the masses, (which has been proved by a great number of observations), whatever may be the nature of the fuel; from whence may be deduced, that the weight of fuel burned in an unit of time, ought

to be exactly proportionate to its density; and in consequence, the densest fuel furnishing the most food for combustion, ought to give out the greatest heat. But, as for the same reason they consume a larger proportion of oxygen, they require, in order to produce all their effect, a more rapid and strong current of air.

It is clear, from that which has been stated concerning the relative properties of coke and charcoal, that when the former can be procured of good quality, and especially when the ash contains but little oxide of iron, it ought to be preferred to charcoal, for assays requiring a high temperature.

A very essential condition in obtaining the maximum effect of a furnace, and the importance of which can be alone appreciated by experience, is the choice of pieces of fuel of a suitable size. If, on the one hand, a shovelful of coke or charcoal, be taken at random, it generally contains the dust and dirt found in most fuel, and which by filling all the interstices, prevents the air from passing as required, and the consequent combustion is slow. On the other, if a furnace be filled with large pieces, considerable spaces are left between them, so that but a comparatively small surface is exposed to the action of the atmospheric oxygen, and a correspondingly small quantity of fuel consumed in a given time, so that the maximum heat can never be obtained. In order to produce this desirable result, it is necessary that the pieces have a certain mean size, and experience has proved that pieces about 1 inch to $1\frac{1}{4}$ inches square, produce the best effect.

THE EFFECTS PRODUCED BY WIND AND BLAST FURNACES.

Assays by the dry way can be made either in wind or blast furnaces. In either of them, the degree of heat depends upon the volume of air which passes through the fuel in the same time; but, *cæteris paribus*, large furnaces produce more heat than small, because comparatively less heat is lost by the first than the last.

In a wind furnace, the maximum of heat is limited by the size of the chimney, and in a blast furnace, by the dimensions of the bellows; but by weighting the latter, more or less, the force of the blast can be increased, and, in consequence, the temperature, to an almost indefinite extent. In this respect, blast have the advantage over wind furnaces.

In the latter, the draught increases in proportion as the heat becomes more intense in the furnace, so that the temperature producible increases progressively. In a blast furnace, the bellows can be weighted and worked as heavily as possible at once, and by opening all the apertures for receiving air, the maximum temperature can be produced nearly immediately, and a very high temperature obtained more rapidly than in a wind furnace; but this is of little use, because, as heat passes very slowly into the substance of a crucible, and when the object is to fuse its contents, it must be heated gradually, without running the risk of softening the crucible before its contents were acted upon, or even scarcely made warm.

Wind furnaces are, however, infinitely more serviceable and economical than blast, because they work themselves, and do not require the service of a man to attend

to the bellows. A blast furnace is useful in a laboratory, in certain cases, for instance, when a single crucible has to be submitted to an intense heat; and when the furnace is small and the bellows large, in which case the operation resembles a blow-pipe assay.

In whatever manner the air is introduced into any kind of furnace, either wind or blast, it is evident that the quantity of heat developed in equal sized furnaces depends upon the quantity of air introduced in the same time; but the degree of temperature is not the same in different parts of the furnace, and the distribution of heat varies according to the manner in which the air is introduced into the midst of the fuel. The side over which the air passes is kept cold by the current, on which account, fire-bars last a long time without becoming oxidated, when the draught is stopped only when the fuel is totally consumed; but the heat rapidly augments up to a certain distance from the bars, at which place it has arrived at its maximum; above that it diminishes rapidly, because the air is nearly deprived of its oxygen. Experiment has proved that this maximum is about $2\frac{1}{2}$ to 3 inches above the bars or tuyères.

In common wind furnaces, the air enters by horizontal bars, which form the bottom of the furnace, and the crucibles are placed on a stand which rests on these bars. By this means, the lower and centre part of the crucibles, and in which parts the matter to be fused is placed, are exactly situated in the maximum of heat, but the stand being constantly kept cold by the contact of a current of air, establishes a continual draining or carrying away of heat from the interior of the crucible

outwards, so that the substance submitted to assay can never arrive at the maximum temperature but after a length of time, and then *that* maximum is always inferior to that in the mass of fuel. It is on this account that assays in a blast or wind furnace generally occupy from one hour to two hours. I have found that the time may be reduced to half that just stated, if a good solid foundation of fuel be made, and the crucible placed on that, and well surrounded by coke constantly kept close to the pot and the sides of the furnace; in this manner the cooling effect of the stand is removed, and the consequent maximum effect of the furnace produced.

The best fire lute is that employed by Mr. Parker, and is composed of good clay 2 parts, sharp washed sand 8 parts, horse-dung 1 part. These materials are to be intimately mixed; and afterwards, the whole is to be thoroughly tempered, like mortar. Mr. Watt's fire lute is an excellent one, but is more expensive. It is made of finely powdered Cornish (porcelain) clay, mixed to the consistence of thick paint, with a solution of borax, in the proportion of two ounces of borax to a pint of hot water.

It may be as well to mention in this part of the work, the various lutes which may be employed, either in fire operations, or in making good joints in experiments with gases or liquids. The following are the principal kinds.

1. *Fat lute* is prepared by mixing dry clay, in a fine powder, with drying oil, so that the mixture may form a ductile paste. When this paste is used, the part to

which it is applied ought to be very clean and dry, otherwise it will not adhere.

2. *Roman cement*, which must be kept in well-closed vessels, and not moistened until the instant it is required for use.

3. *Plaster of Paris*, mixed with water, milk, or weak glue, or starch water. These three lutes stand a dull red heat. The two latter may be rendered perfectly impermeable to gaseous bodies by being smeared over with oil, or a mixture of oil and wax.

4. *Linseed or almond meal*, mixed to the consistence of a paste with water, milk, lime-water, or starch paste. This lute is very manageable and impermeable, but does not withstand a heat greater than about 500°.

5. If just the sufficient quantity of water be added to quick-lime to reduce it to a dry powder, and that is mixed well and rapidly with white of egg diluted with its own volume of water, and the mixture spread immediately in strips of linen and applied to the part, then powdered with quick-lime, it forms a good cement. Instead of white of egg, lime and cheese may be used, or lime with weak glue water or blood. This lute dries very rapidly, becoming very hard, and adhering strongly to the glass; but its great inconvenience is the want of flexibility.

6. *White lead mixed with oil.*—If the mixture be spread upon strips of linen, or bundles of tow, it acts much in the same manner as the lime lutes.

7. *Yellow wax* is often used as a lute, but it becomes very brittle at a low temperature. It may be rendered

less brittle, and at the same time more fusible by an admixture of one eighth of crude turpentine.

8. *Soft cement* is prepared by fusing yellow wax with half its weight of crude turpentine and a little Venetian red, in order to colour it. It is very flexible, and takes any desired form under the pressure of the fingers.

9. *Resinous, or hard cement,* is made by fusing together at the lowest possible temperature 1 part of yellow wax and 5 or 6 of resin, and then adding gradually 1 part of red ochre, or finely powdered brick-dust, (plaster of Paris succeeds very well), and then raising the temperature to 212° at least, until no more froth arises, or agitation takes place, and stirring it continually until cold. This cement is employed in a hot state. This lute is much used for fixing brass caps, &c., to air jars.

10. *Paper*, covered with common glue, is occasionally employed.

11. *Bladders* cut in small strips are occasionally used in covering other lutes when the pressure of gas is considerable, or when the lute is subject to strain from any other cause. They are digested in water until they become soft and flaccid, they are then applied to the part like a piece of pasted paper by the pressure of the hand. These strips adhere very strongly to glass or earthenware, and their adhesive power may be much augmented by smearing them with white of egg. Lastly, the joints made in this manner may be made firmer by binding them with string or fine wire.

12. *Caoutchouc.*—Tubes of this material form a very ready means of attaching one piece of apparatus to

another, and they possess the peculiar advantage of flexibility, which allows the various parts of the apparatus which they connect to move in different directions to a slight extent, so that the whole is not so likely to be fractured as when connected in an inflexible manner. Caoutchouc is also less acted upon by gases and vapours than almost any other substance we know; even chlorine attacks it but slowly, and it possesses the valuable property of forming a perfect joint when freshly cut joints are brought and pressed together; hence the facility with which it is manufactured into tubes. The mode of manufacture is as follows: Take a piece of the sheet caoutchouc of the required size, and warm it either in the hand, or before a fire until it is perfectly soft, then place it around a glass rod of the requisite size, pressing the edges close together with the fingers; when close together, cut off the superabundance with a sharp pair of scissors, and the newly cut edges will unite by simple pressure of the nail. When well executed, the join is scarcely apparent. In order to prevent the caoutchouc from adhering to the rods on which the tube is formed, a little moisture or starch powder may be employed.

When caoutchouc is not at hand, oiled paper may be substituted, the joint being made with wax.

Faraday gives the following directions for luting iron, glass, or earthenware retorts, tubes, &c., for furnace operations. When the lute has to withstand a very high temperature, it should be made of the best Stourbridge clay which is to be made into a paste, varying in thickness according to the opinion of the operator. The paste

should be beaten until it is perfectly ductile and uniform, and a portion should then be flattened out into a cake of the required thickness, and of such a size as shall be most manageable with the vessel to be coated. If the vessel be a retort or flask, it should be placed in the middle of the cake, and the edges of the latter raised on all sides, and gradually moulded and applied to the glass; if it be a tube, it should be laid on one edge of the plate, and then applied by rolling the tube forward. In all cases, the surface to be coated should be rubbed over with a piece of the lute dipped in water, for the purpose of slightly moistening and leaving a little of the earth upon it; if any part of the surface becomes dry before the lute is applied, it should be re-moistened. The lute should be pressed and rubbed down upon the glass successively from the part where the contact was first made to the edges until all air bubbles are excluded, and an intimate adhesion effected. When one cake of lute has been applied, and is not large enough to cover the whole required surface, another must be adapted in a similar manner. Great care must be taken in joining the edges, for which purpose it is better to make them thin by pressure, and also somewhat irregular in form, and if at all dry they should be moistened with a little soft lute. The general thickness may be about one quarter to one third of an inch.

Being thus luted, the vessels are afterwards to be placed in a warm situation, over the sand-bath or near the ash-pit, or in the sun's rays. They should not be allowed to dry rapidly or irregularly, and should be moved now and then to change their positions.

To prevent cracking during desiccation, and the consequent separation of the coat from the vessel, some chemists recommend the introduction of fibrous substances into the lute, so as mechanically to increase the tenacity of its parts. Horse-dung, chopped hay and straw, horse and cow-hair, and tow cut short are amongst the number. When they are used, they should be added in small quantity, and it is generally necessary to add more water than with simple lute, and employ more labour to ensure an uniform mixture. It is best to mix the chopped material with the clay before the water is put to it, and by adding the latter to effect the mixture, at first by stirring up the mass lightly with a pointed stick or fork; it will then be found easy, by a little management, to obtain a good mixture without making it very moist.

The luting ought to be made as dry as possible, consistent with facility in working it. The wetter it is, the more liable to crack in drying, and *vice versâ*.

Mr. Willis recommends when earthenware retorts, &c., are to be rendered impervious to air the following coating. One ounce of borax is to be dissolved in half a pint of boiling water, and as much slaked lime added as will make a thin paste. This composition is to be spread over the vessel with a brush, and when dry, a coating of slaked lime and linseed oil is to be applied. This will dry sufficiently in a day or two, and is then fit for use.

Iron cement. — This mixture is used for making permanent joints generally between surfaces of iron. Clean iron borings or turnings are to be slightly pounded so as to be broken but not pulverized; the

result is to be sifted coarsely, mixed with powdered sal-ammoniac and sulphur, and enough water to moisten the whole slightly. The proportions are, 1 sulphur, 2 sal ammoniac, and 80 iron. No more should be mixed than can be used at one time.

CRUCIBLES, CUPELS, &c.

The crucibles best known in commerce are the Hessian, the Cornish, and the London; the latter of which are much to be preferred, on account of their general refractory nature; they are also much better made than the two other kinds, being much smoother and more regularly formed. They have the form of a triangular pyramid, and are made in such sizes that they fit one into the other, forming nests. The triangular form is very convenient, because there are three spouts, from either of which can be poured the fused contents of the pot. The Cornish crucibles are circular, and do not stand changes of temperature so well as the London pots, neither can they endure such an extreme of heat, for they agglutinate and run together at a temperature which does not touch the others. The Hessian pots are worst of all, they do not stand the least change of temperature without a certain fracture, so that they require to be very carefully used. There is also another kind of pot in use, made of the same material as the London crucibles, termed a "skittle pot," from its resemblance, no doubt, to the ordinary wooden skittle or nine-pin. They are exceedingly useful for the fusion of large masses of matter, or such substances as boil or bubble much when heated.

Crucibles, in order to be perfect and capable of being used indifferently for any operation, ought to possess the four following qualities: Firstly, not to break or split when exposed to sudden changes of temperature; secondly, to be infusible; thirdly, to be only slightly attacked by the fused substances they may contain; fourthly and lastly, to be impermeable, or nearly so, to liquids and gases. But as it is very difficult to unite all these qualifications, various kinds of pots are made to fulfil one or more of them.

In order to render crucibles capable of withstanding changes of temperature without breaking, a certain proportion of substances, infusible by themselves, is mixed with the pasty clay; sand, flint, fragments of old crucibles, black-lead and coke are used for this purpose. They are reduced to a state of division more or less fine, according to the grain of the clay paste. For ordinary pots, the powder ought not to be very fine; but for porcelain crucibles it ought to be as fine as flour. The choice of these various bodies depends upon the use for which the crucible is intended.

The most refractory crucibles are those made with the pure clays, or such as contain little or no oxide of iron, and especially free from calcareous matters. Amongst those clays, the best are those which contain most silica; nevertheless, crucibles of pure clay are not absolutely infusible, and in the high temperature of a wind furnace, they sometimes soften so much as actually to fall into a shapeless mass. This defect, as before stated, can be in some measure diminished by mixing with the clay, a quantity of graphite or coke; either of these substances form a kind of solid skeleton,

which retains the softened clay, and prevents its falling out of shape.

Coke and black-lead are more efficacious than sand, because they have no action on clay, and because quartz forms a fusible compound with it. If too large a quantity of black-lead or coke be employed, it gradually consumes in the fire, and the pots become porous, and break at the least movement. Wood charcoal can be used instead of black-lead or coke, but is not so good because it burns more readily.

Black-lead crucibles are generally composed of 1 part of refractory clay, and from 2 to 3 of black-lead. These pots withstand all possible changes of temperature without cracking, and their form is rarely changed by the heat, not because they are absolutely infusible, but because they are supported by the skeleton of graphite.

Black-lead being a very rare and expensive material, certain artificial mixtures have been contrived, coke seems to be the best substitute. Those made by Marshall and Anstey are said to approach, in many of their properties, to the ordinary blue pots. They are made of Stourbridge clay and pulverized coke; about 2 parts of the former and one of the latter.

Crucibles into whose composition carbonaceous matters enter, reduce any oxides that may be heated in them, and hence are inconvenient in certain cases. They can, nevertheless, be employed in all cases, by giving them a lining of clay, which must be tolerably thick and well dried before use.

Earthen crucibles, which have not been baked at a white heat, are more or less permeable to liquids and

gases, according to the grain. In order to render them impermeable to liquids, they must be heated to such a temperature as will suffice to fuse the outside. When treated in this way, however, they are very liable to crack with sudden changes of temperature; the best method, therefore, of rendering them capable of containing water, &c., is to coat them with the mixture of borax and lime, described as Willis's lute.

Resistance to Corrosion. — In order that crucibles may resist the corrosive action of the fused substances contained within them, they must be as compact as possible, and the substance of which they are made must have little or no tendency to combine with the fused contents. The metals and their non-oxidated compounds neither attack clay nor black-lead; but there are, nevertheless, some metallic substances, galena, for instance, which without exercising any chemical action on earthy matters, have the property of filtering through their pores.

The readily reducible oxides gradually corrode black-lead crucibles, and those pots in the composition of which coke enters by burning the carbonaceous matter. The greater portion of these same oxides, the alkalies, earths, and glasses, which are the fusible silicates, borates, &c., act more or less powerfully on the earthy base of all crucibles, so that these substances are the most difficult to keep in fusion for any length of time. They attack the crucible layer by layer, dissolving the substance of which it is composed, and after a lapse of time, rendering it so thin that it cannot withstand the pressure of the molten mass within it, and the fracture of the pot, and consequent loss of contents is inevitable.

Impermeability.—Under the same circumstances, all those crucibles whose texture is loose, are more readily corroded than those with a firm compact body, because the corrosive substance filters to a certain depth in the former crucibles, and, in consequence, has a larger surface to act upon than when it is contained in a compact pot.

Examination of Crucibles.—Earthen crucibles may be assayed by the time they will contain fused litharge, which exercises a very corrosive action, honeycombing them in all directions, and those pots which contain it longest without undergoing much damage, may be considered the best. However, this method of assay is not exact, even by taking into account the thickness of the pot, for litharge runs through crucibles; firstly, because it is very fusible and easily filters through their pores; and secondly, it has the property of forming fusible compounds with all the silicates by combining with them. From these remarks, it will be evident that a crucible whose grain is loose, will readily allow litharge to pass through it, however slightly its substance may be fusible or acted on; or if, on the contrary, it be very easily acted on, even when absolutely infusible, with an extremely fine grain: the promptitude with which a crucible is traversed by litharge, bears no relation to its fusibility. A crucible of pure quartz will be very readily attacked by litharge, because it has much affinity for silica; and the simple silicates of lead are all very fusible, whilst a crucible composed of silica, alumina, and lime, which by itself is very fusible, would be corroded less rapidly, because the oxide of lead has much less affinity for the

earths than it has for the silica; moreover, it forms less fusible compounds with the earths than with silica alone. The assays of crucibles made with litharge, if not of use in ascertaining their degree of fusibility, fulfils perfectly its object when it is wished to prove the resistance a crucible has to the corrosive action of various bodies in a state of fusion; for of all fusible substances, none exercise such a powerful action on earthy matters as litharge.

Crucibles ought not only to resist the corrosive action of those bodies they may contain, but also that of the ash produced by the combustion of the fuel in which they may be placed. These ashes being often calcareous, alkaline, or ferruginous, act on the clayey part of the crucibles exactly as the fluxes. From whence it follows, that those crucibles which contain litharge longest, will also resist the action of the fluxes best.

In order to ascertain the fusibility of a crucible, a direct experiment must be made, either by heating a piece in a crucible lined with charcoal, and ascertaining if its angles be rounded, if its substance has become translucid, &c.; or better still, by heating the crucible to be assayed with another whose properties are well known.

As to permeability, it may approximately be ascertained by filling two crucibles with water, and noting how long it takes to completely empty them by transudation; the crucible which contains it longest is, of course, the least permeable.

To prove if a crucible be able to sustain great changes of temperature without breaking, introduce it, perfectly cold, into a furnace full of lighted coal, take it out when

reddish white, and expose it to a current of cold air produced by a bellows or otherwise; if it stand these trials, it may be heated afresh and plunged red hot into water, and if it be not broken, placed immediately in the fire. The best pots support all these operations without breaking; but it often happens that they are filled with innumerable small fissures, through which fused matters can pass. This can be ascertained by fusing rapidly in the assay pot a quantity of litharge; if these be present, the fused oxide will readily filter through them.

Charcoal Crucibles.—As all oxidated matters act very readily on clay pots, and a great number of the metals and their compounds adhere to them, they have long since been replaced, under certain circumstances, by charcoal crucibles, which do not possess these disadvantages. The older assayers used merely a piece of charcoal, with a hole made in it, and then bound round with iron or other wire. The use of these has, however, been abandoned for some length of time, and earthenware crucibles lined with charcoal have been substituted. These may be considered as charcoal pots enveloped with refractory clay; they are solid, always free from cracks, and easy of preparation, and they have the same properties as the solid charcoal crucibles, without their inconveniences.

In order to prepare these crucibles, the charcoal must be chosen carefully, so as to contain no foreign substances; it must be pulverized and passed through a sieve, the powder moistened with water, mixed with a spatula, and then kneaded with the fingers until it just

adheres, and forms cohesive lumps without being sufficiently wet to adhere to the hand. Some advise the addition of gum to the water with which the charcoal is moistened; which addition is, however, useless, water alone being sufficient to give a suitable consistence to the charcoal lining. Crucibles are lined with charcoal by the following method.

The crucible is moistened slightly by being plunged into water and withdrawn as speedily as possible, and about ½ an inch in depth of the charcoal paste, prepared as above, placed in it; the paste is then pressed firmly down by means of a wooden pestle; the blows are to be slight at first, and then increasing in force until it is as firm as possible; another layer is then applied and pressed as before, and the process repeated until the crucible is quite full, taking great care to render all as firm as possible, especially at the sides. In order to make each layer adhere firmly to the other, they must be scratched rather deeply with the point of a knife before a new layer is applied. When the crucible is completely filled, a hole is to be scooped in the charcoal, of about the form of the pot. The sides are then rendered smooth by friction with a glass rod. This is absolutely necessary, so that the metallic globules produced in an assay may not be retained by the asperities of the lining, but may be readily enabled to unite into one button. When a lined charcoal pot is well made, its sides are very smooth and shining. For ordinary use, the lining may be ⅝ths of an inch thick at the bottom, and ¼th or so at the sides; but in some cases, for instance, when the substance to be fused is capable of filtering through the lining and attacking the pot, as a flux, it

must be at least twice the above thickness in every part.

As before stated, in our article on Reduction, lined crucibles have many advantages over plain crucibles. The lining gives them greater solidity, and prevents a loss of shape when softened; for plain crucibles are always three-fourths empty when their contents are fused; on account of its diminishing much in volume, the pots then have nothing to sustain their sides at the time they soften towards the end of the assay, when the highest temperature is employed. Besides, glassy matters do not penetrate the lining, and exercising no action on it, can be obtained in a state of purity, and their exact weight ascertained; if they were fused in a plain pot, they could not be weighed, because a portion would adhere to the sides, and they would not be pure, having taken up a portion of the crucible in which they were fused.

The lining, too, effects the reduction of certain metallic oxides by cementation, and does away with the necessity of adding powdered charcoal to the body to be reduced. This property is very valuable, because, when an oxide is reduced by mixing it with charcoal, an excess must always be employed, and which excess remains with the metal, and prevents us from obtaining its exact weight.

In certain particular experiments, crucibles are lined with other bodies besides charcoal, such as silica, alumina, magnesia, or chalk, by merely moistening their respective powders with water, and applying the paste as above described for the charcoal. A slight layer of chalk lessens the liability of attack from fused litharge.

Malleable iron crucibles are often very serviceable in assays of fusibility, and of certain seleniurets and sulphurets. They are either made of hammered sheet-iron, or by plugging up small iron tubes, as gun-barrels, &c. The latter are preferable, because thick, solid crucibles can be used a number of times, whilst the others are necessarily very thin, and can be used only once. Whenever iron crucibles are employed at a very high temperature, they must be placed in those of earthenware, which protect them from the oxidating action of the air; but when they are not heated above the temperature of a copper assay, they may be used naked, if they are tolerably thick.

For assays at the above temperature, cast iron crucibles may be employed with advantage, instead of wrought iron, because they are very nearly as good, and much less expensive.

CUPELS

are vessels in which the operation termed cupellation is carried on. They are made of such substances as are not acted upon by certain fused oxides, as those of lead or bismuth, and their texture is sufficiently loose to allow those oxides to penetrate their substance readily, and yet be sufficiently strong to bear handling without breaking.

There are a great number of substances of which cupels can be made, which will fulfil all these conditions, but only one is in general use, viz.: the ash of burnt bones. The powdered and sifted ash is washed repeatedly with water, to remove all saline and extraneous soluble matter; and, lastly, dried. It now consists

principally of pure phosphate of lime, with a little carbonate partially decomposed. It is sometimes made into a paste with water, but I have found beer to answer much better. The following are the proportions I employ: 4 lbs. of bone-ash, and ½ lb. of beer. The above mixture is just sufficiently moist to adhere strongly when well pressed, but not so moist as to adhere to the finger or the mould employed to fashion the cupels. The mould consists of two pieces, one a ring, having a conical opening; the other, a pestle, having a hemispherical end fitting the larger opening of the ring. In order to mould the cupels, proceed as follows: Fill the ring with the composition, then place the pestle upon it, and force it down as much as possible; by this means, the moistened bone-ash will become hardened, and take the form of the pestle; the latter must then be forced as much as possible, by repeated blows from a hammer, until quite home. It is then to be turned lightly round, so as to smooth the inner surface of the cupel, and withdrawn; the cupel is removed from the mould by a gentle pressure on the narrowest end. When in this state, the cupel must be dried gently by a stove; and lastly, ignited in a muffle, to expel all moisture. It is then ready for use.

There are two or three points to attend to in manufacturing the best cupels. Firstly, the powdered bone-ash must be of a certain degree of fineness; secondly, the paste must be neither too soft nor too dry; and thirdly, the pressure must be made with a certain degree of force. A coarse powder, only slightly moistened and compressed, furnishes cupels which are very porous, which break on the least pressure, and which

allow small globules of metal to enter into their pores, which is the most serious inconvenience of all.

When, on the contrary, the powder is very fine, the paste very moist and compressed very strongly, the cupels have much solidity, and are not very porous, the fine metal cannot penetrate them, and the operation proceeds very slowly; besides, the assay is likely to become dulled and incapable of proceeding without a much higher degree of temperature being employed.

METHODS OF MEASURING THE HEAT OF FURNACES.

As much of the accuracy of an assay depends on the temperature at which it is made, and the temperature required varies with the metal, it is very desirable to possess some means of ascertaining the heat of the furnace more accurately than by the eye. There have been instruments devised for this purpose by several parties. The chief inventors are Mr. Wedgwood and the late Professor Daniell, of King's College. The instruments are termed pyrometers, and both of those just mentioned will be described, and their peculiarities and use pointed out, commencing with Wedgwood's.

This pyrometer is constructed on the principle that the purest clay is contracted in proportion to the heat applied to it. This contraction is occasioned by its giving up the water which it holds with great tenacity, by exposure to a high temperature. It is composed of two parts; of a gauge which serves to measure the degrees of heat, and of small pieces of clay which are employed to ascertain the same degrees by their con-

traction. The gauge is formed of a plate of brass, with two rulers of the same substance firmly fixed to it. The two rulers are 24 inches long, and fixed in the plate $\frac{5}{10}$ths of an inch asunder at one end, and $\frac{3}{10}$ths of an inch at the other; so that the distance between the two rods gradually diminishes, and the whole diminution amounts to $\frac{2}{10}$ths of an inch. The ruler is divided into 240 equal parts, each of which measure $\frac{1}{10}$th of an inch.

To form the pieces by whose contraction the heat is measured, the finely powdered and sifted clay is mixed with water, and the paste made to pass through an iron tube; it is then cut into cylinders of a suitable length. When these pieces are dry they must be adapted to the zero of the gauge. When this pyrometer is used, one of the pieces of clay is placed in the furnace whose temperature is to be ascertained, and when it has experienced the utmost extremity of the heat, it is withdrawn and allowed to cool. Then it is placed in the gauge, and passed between the two rulers until it will go no further, the degree of heat is then calculated from the contraction which it has undergone.

The following is a table drawn up by Mr. Wedgwood of some of the corresponding degrees of his pyrometer with those of the Fahrenheit thermometer.

	Wedgwood.	Fahrenheit.
Red heat, visible by daylight	0^0	1077^0
Copper melts	57	4587
Silver melts	28	4717
Gold melts	32	5237
Cast iron melts	130	17977
Greatest heat of wind furnace	160	21877

The indications of this pyrometer are, however, very inaccurate, from the fact that clay will contract as much by the long continuance of a low heat, as by the short continuance of a high one. Hence, the degrees of heat measured by Wedgwood's pyrometer have been enormously exaggerated. It was long since noticed that it does not produce comparable effects, and this was supposed to proceed wholly from the impossibility of obtaining clay perfectly alike for each experiment.

This led M. Guyton* to propose another form of pyrometer, which is essentially the same as Professor Daniell's. The only difference in the latter is its more perfect construction. It is composed of a rod of platinum simply laid in a groove made of refractory clay, and baked in the highest degree of heat. This rod rests at one end on the edge which terminates the groove, and at the other on a lever with two arms, the larger of which forms a needle on a graduated arc of a circle, so that the removal of this needle, from its position, marks the additional length which this metal acquires by the heat. I will now give Professor Daniell's description of his own pyrometer; and the resemblance between the two will be at once seen.

"It consists of two parts, which may be distinguished as the register and the scale. The register is a solid bar of black-lead earthenware highly baked. In this a hole is drilled, into which a bar of any metal, six inches long, may be dropped, and which will then rest upon its solid end. A cylindrical piece of porcelain, called the index, is then placed upon the top of the bar, and confined in its place by a ring or strap of

* Annales de Chimie, vol. XLVI. p. 276.

platinum passing round the top of the register, which is partly cut away at the top, and tightened by a wedge of porcelain. When such an arrangement is exposed to a high temperature, it is obvious that the expansion of the metallic bar will force the index forward to the amount of the excess of its expansion over that of the black-lead, and that when again cooled it will be left at the point of greatest elongation. What is now required, is the measurement of the distance which the index has been thrust forward from its first position, and this, though in any case but small, may be effected with great precision by means of the scale."*

This is independent of the register, and consists of two rules of brass accurately joined together at a right angle by their edges, and fitting square upon two sides of the black-lead bar. At one end of this double rule, a small plate of brass projects at a right angle, which may be brought down upon the shoulder of the register formed by the notch cut away for the reception of the index. A moveable arm is attached to this frame, turning at its fixed extremity on a centre, and at its other carrying the arc of a circle, whose radius is exactly five inches, accurately divided into degrees, and thirds of a degree. Upon this arm, at the centre of the circle, another lighter arm is made to turn, one end of which carries a nonius with it, which moves upon the face of the arc, and subdivides the former graduation into minutes of a degree; the other end crosses the centre and

* Daniell's Chemical Philosophy, p. 111.

terminates in an obtuse steel point, turned inwards at a right angle.

When an observation is to be made, a bar of platinum or malleable iron is placed in the cavity of the register; the index is to be pressed down upon it, and firmly fixed in its place by the platinum strap and porcelain wedge. The scale is then to be applied by carefully adjusting the brass rule to the sides of the register, and fixing it by pressing the cross piece upon the shoulder, and placing the moveable arm so that the steel part of the radius may drop into a small cavity made for its reception, and coinciding with the axis of the metallic bar. The minute of the degree must then be noted which the nonius indicates upon the arc. A similar observation must be made after the register has been exposed to the increased temperature which it is designed to measure, and again cooled, and it will be found that the nonius has been moved forward a certain number of degrees or minutes. The scale of this pyrometer is readily connected with that of the thermometer by immersing the register in boiling mercury, whose temperature is as constant as that of boiling water, and has been accurately determined by the thermometer. The amount of expansion for a known number of degrees is thus determined, and the value of all other expansions may be considered as proportionate.

We shall now see, by comparing the indications as determined by this pyrometer with those of Wedgwood, to what an enormous extent those of the latter were wrong.

By Daniell's, the melting point of cast iron has been

ascertained to be 2786°, and the highest temperature of a good wind furnace, 3300° Fahrenheit, points which, as the table before mentioned, show were estimated by Mr. Wedgwood at 17977° and 21877° respectively.

The following is a list of the melting points of some of the metals, and it is obvious that in an assay of each particular metal, the temperature employed must exceed by a considerable number of degrees its melting point. The table is, therefore, very useful.

	Fahrenheit.
Tin melts at	422°*
Bismuth	497
Lead	612
Zinc	773
Cadmium	442
Silver	1860
Copper	1996
Gold	2016
Cast iron	2786

Cobalt and nickel rather less fusible than iron.

* Daniell.

CHAPTER III.

THE FLUXES, THEIR COMPOSITION, MODE OF PREPARATION AND USE.

In some of the operations in the dry way, bodies are heated in suitable vessels *per se*; but more often it is necessary to add to the bodies submitted to assay, other substances which are varied according to the nature of the change to be effected. As these substances generally determine the fusion of the body to which they are added, they are termed fluxes or fusing agents; but this name cannot be applied to all of them with exactitude.

They are generally divided into five classes:—1. reducing agents; 2. oxidating agents; 3. desulphurating agents; 4. sulphurating agents; and lastly, fluxes properly so called.

REDUCING AGENTS.

All the substances belonging to this class have the power of removing oxygen from those bodies with which it may be combined. They are as follows:

1. Hydrogen gas.
2. Charcoal.
3. The fat oils, tallow, and resins.
4. Sugar, starch, and gum.

5. Tartaric acid.
6. Oxalic acid.
7. Metallic iron, and lead.

Hydrogen Gas.—This body is so termed, because in combining with oxygen it forms water. The most common method of preparing this gas consists in dissolving zinc in dilute sulphuric acid. It is invisible and colourless when absolutely pure, and is the lightest body in nature. It is a most powerful reducing agent, and reduces a great number of metallic oxides at a red or white heat, viz. the oxides of lead, bismuth, copper, antimony, zinc, iron, cobalt, nickel, tungsten, molybdenum, and uranium. When any metal is required in a state of absolute purity, this is the only reducing agent admissible, as all others give the metal combined with a certain proportion of charcoal.

Carbon.—Perfectly pure carbon is exceedingly rare in nature. It is found in large quantities in the mineral kingdom combined with other bodies. In a perfect state of purity it constitutes the diamond. The diamond, like all other species of carbon, is unacted on by the highest possible temperature when in close vessels. It burns in atmospheric air and oxygen gas, but requires a higher temperature than ordinary charcoal. After the diamond the most remarkable and purest species of carbon are:

Firstly. *Black-lead* or *Graphite*, this is a mineral found in beds in the primitive formations, principally in granite and mica-schist. The purest at present known is found at Borrowdale in Cumberland.

Secondly. *Anthracite* is another species of fossil carbon much resembling ordinary coal, but differing from it by burning with neither smell, smoke, or flame.

Thirdly. *Coke* is the residue of the coal employed in the gas works after all the volatile matter is expelled. It is generally iron black, and has nearly a metallic lustre: it is difficult to inflame, and burns but in small pieces, but gives a very intense heat.

Fourthly. *Wood Charcoal* is obtained by burning the woody part of plants with a limited supply of air, so as to drive off all their volatile matters, and leave merely their carbon. It is this kind that is generally employed in assays. It ought to be chosen with care, well pulverized, passed through a sieve and preserved in well stopped vessels. Wood charcoal is never perfectly pure, it generally contains a proportion of hydrogen and watery vapour; these bodies are not exactly prejudicial, but in some experiments they ought not to be present; in that case pure charcoal may be readily procured by heating sugar to redness in a close crucible.

Charcoal by itself possesses two inconveniences: firstly, it has the property of combining with many metals; and in the second place it is infusible, and cannot combine with vitreous substances. The property it possesses of combining with iron, nickel, cobalt, &c., is of no consequence to the assayer, for the increase of weight it gives is not material; but its infusibility and inability to combine with fluxes is a very serious inconvenience; for after the reduction, that portion which has not been consumed, remains disseminated with the grains of metal in the fused slag, and

prevents the separation of all the metal, and the consequent formation of a good button; a large quantity of charcoal can thus irreparably injure an assay. This inconvenience does not happen, however, when an oxide is reduced by cementation in a lined crucible; but there are some cases in which its employment is inadmissible.

In default of charcoal, coke may be employed, but it must be chosen with much care, for it often contains a very large proportion of earthy and other extraneous matters; so that before use, it is necessary to burn a quantity, in order to ascertain their amount and composition, and that coke ought only to be used which gives nearly white ashes, and which contains no more than a few per cents. Coke is never so good as wood charcoal as a reducing agent, because it burns more slowly by combining less rapidly with oxygen. When it is used the temperature employed for an assay must be much increased.

Coal is nearly always inconvenient, because it swells by heat; nevertheless as it is not required in very large quantity it is sometimes employed, being previous to use finely powdered and sifted.

THE FAT OILS, TALLOW AND RESIN.

The Fat Oils.—The name oil is generally given to those bodies that are fat and unctuous to the touch, more or less fluid, insoluble in water, combustible and forming soaps with alkalies. They all congeal and become solid at various degrees of temperature. There are even some which, in the temperature of our climate,

have constantly a solid form, as butter, palm oil, cocoa nut oil, &c.

Fixed oils have a very marked unctuosity, they neither dissolve in water nor alcohol, and take fire at a heat capable of reducing them to a state of vapour. Their density is from .915 to .940, and the boiling temperature about 600°. When distilled, they are decomposed, disengaging margaric and oleic acids, &c., then an empyreumatic oil, and lastly a yellowish red substance, and leaving about 2 per cent. of charcoal. When heated to a higher temperature than their boiling point, they are very nearly wholly resolved into an inflammable gas.

According to MM. Gay Lussac and Thénard, these oils consist of:

Carbon . . .	.77213
Oxygen . . .	.09451
Hydrogen . . .	.13336
	100000

Tallow is an animal product analogous to the fat oils both in chemical composition and properties; it is soft but solid, white, translucid and granular. According to Chevreuil, mutton fat is composed of:

Carbon . . .	.78876
Oxygen . . .	.11790
Hydrogen . . .	.09334
	100000

The Resins.—The greater part of the resins are solid; but some are soft. They are brittle with a vitreous and shining fracture, and often transparent. They are very

fusible, but cannot be raised to their boiling point without partial decomposition. They give an acid water, an empyreumatic oil, and combustible gas by distillation, leaving a variable proportion of charcoal.

Colophony has the following composition:

Carbon . . .	75944
Oxygen . . .	13338
Hydrogen . . .	10718
	100000

Although all the bodies just mentioned consume in their combustion a large quantity of oxygen, they cannot generally effect the total reduction of an oxide, because of their volatility; so that before the temperature at which the reduction takes place, can be attained, the greater part of the reducing agent has been expelled. They generally act only by virtue of the small carbonaceous residue produced by the action of heat, so that their use is very limited and uncertain. Whenever they are employed as reducing agents, without covering the substance, a loss can be experienced, on account of the bubbling and boiling caused by their decomposition; this will always take place, unless the contents of the crucible be covered with charcoal powder. Oils are very serviceable in the reduction of a large mass of oxide by cementation; in this case, after the oxide has been placed in the crucible, as much oil is added as the oxide, and the lining of the crucible will soak up. Fat or resin is also used to prevent the oxidation of the surface of a metallic bath, by reducing the film of oxide formed by the action of the atmospheric oxygen.

SUGAR, STARCH, AND GUM.

These three bodies are so well known that a description is useless, so their use will merely be mentioned.

All three have the same chemical composition, viz.

Carbon	44.91
Hydrogen . . .	6.11
Oxygen . . .	48.98
	100.00

Sugar in its decomposition by heat leaves a much larger proportion of carbon than the oils, fats, or resins; so that it would appear serviceable as a reducing agent. There are some cases in which it may be used with advantage, but unfortunately it undergoes a great increase in volume when heated, and is much agitated at the same time, so that losses in an assay may occur by the use of this agent. The charcoal of sugar is pure carbon, because it leaves no residue when burnt; it is, therefore, preferable to wood charcoal, in cases where no foreign matter can be introduced into the assay.

Starch.—Common starch well dried, and better still torrefied is employed with advantage as a reducing agent, and is much preferable to sugar, as it neither fuses, swells up, or spirts, and in many cases is even preferable to charcoal, because it is in such a fine state of division that it can be more readily and intimately mixed with the substance to be reduced.

Gum.—Decrepitates slightly by heat, softens, agglomerates and boils, without spirting. The gums can be employed as reducing agents under the same

circumstances as sugar and starch, but the two latter are preferable, because they contain no earthy substances.

Tartaric Acid.—When heated in close vessels, it fuses and bubbles very strongly, and decomposes, giving off combustible gases, leaving a little charcoal. It burns when heated in contact with air, giving rise to a peculiar and not unpleasant odour. This acid is the reducing agent in the cream of tartar, or argol, of which so frequent use is made: but the acid is never employed by itself.

Oxalic acid.—Fuses at a temperature of 208° without decomposing, but when heated to 230° it is decomposed, giving rise to 6 parts of carbonic acid, 5 parts of carbonic oxide, and a little formic acid vapour; and when heated strongly, some portions are volatilized without decomposition; it never leaves a carbonaceous residue.

The anhydrous acid consists of:

Carbon . . .	.3376
Oxygen . . .	.6624
	10000

The crystallized acid consists of:

Carbon . . .	1904
Oxygen . . .	3811
Water	4285
	10000

The property which oxalic acid possesses of not leaving a residue would render it remarkably valuable for the reduction of the metallic oxides in cases where

the slightest trace of carbon is to be avoided, if its reducing power were greater; but as it decomposes at a low temperature, and in burning absorbs but a small quantity of oxygen, especially when it has not been dried, so that even for the most easily reduceable oxides a large proportion must be employed. When it is combined with a base, as potash in the salt of sorrel (binoxalate of potash) its reducing power is much augmented, and it is rendered much less volatile.

Oxalate of Ammonia.—When heated in close vessels, it is decomposed and furnishes oxamide. The reducing power is nearly double that of oxalic acid.

Comparative Reducing Power of the above Fluxes.—In order to give an idea of the comparative reducing power of the fluxes just described, I will give the result of some assays made on them by Berthier, by means of litharge.

By heating the same weight of each reducing agent with an excess of litharge, buttons of lead were obtained, where weights were proportional to the quantity of oxygen absorbed, and by comparing them with each other, the reducing power of each flux is given; by taking for unity the weight of the re-agent, calculation has proved that 1 part of pure carbon produces from litharge 34.31 of lead. The following are the results of some experiments:

Pure carbon . . .	34.31
Hydrogen . . .	104.00
Calcined wood charcoal .	31.18
Ordinary wood charcoal .	28.00
Animal oil . . .	17.40

Resin	14.50
Amber resin . . .	30.00
Tallow	15.20
Sugar	14.50
Common starch . .	11.50
Torrefied starch . .	13.00
Gum arabic . . .	11.00
Tartaric acid . . .	6.00
Oxalic acid . . .	.90
Oxalate of ammonia . .	1.70

It must be borne in mind, that these numbers do not represent the quantities of oxygen each re-agent would absorb in complete combustion; but that it only indicates the quantity of metal produced from those oxides reduced about the same temperature as oxide of lead.

In assaying, however, it is rarely that these agents are used by themselves: they are generally mixed with a flux properly so called, and under the head of reducing fluxes, they will be more particularly described.

Metallic Iron.—This metal removes oxygen from the oxides of lead, bismuth, copper, &c., but is rarely added for that especial purpose; and when it does produce this effect, it is generally secondary, because it previously existed in the matter subjected to assay, or was added for some other purpose.

Metallic Lead.—Reduces but a very small number of oxides, but it reduces many to the minimum of oxidation; it also decomposes some sulphates and arseniates.

OXIDATING AGENTS.

The oxidating agents in general use, are as follows :

1. The oxygen of the air.
2. Litharge and ceruse.
3. Silicates and borates of lead.
4. Nitrate of potash.
5. Nitrate of lead.
6. Peroxide of manganese.
7. Peroxide of copper.
8. Peroxide of iron.
9. The caustic alkalies.
10. The alkaline carbonates.
11. The sulphates of lead, copper, and iron.
12. Sulphate of soda.

Oxygen has never yet been obtained as a liquid or solid, although some of the recent experiments of Faraday tend to prove that it may yet be liquified, if not solidified. Its general form, however, is gaseous; and it is under this form it is contained in the atmosphere, of which it constitutes one-fifth. In order to obtain the gas, we cannot act directly on the atmosphere, but must make use of certain metallic oxides, which are reducible by heat, as the oxide of mercury or peroxide of manganese. Oxygen gas has neither smell nor taste, and is about one-tenth heavier than atmospheric air. It has the property of forming compounds with nearly every body, and its affinities are very energetic.

Atmospheric air consists of four-fifths nitrogen, and one-fifth oxygen. It acts on bodies in the same manner as oxygen, but much less energetically.

LITHARGE AND CERUSE.

Litharge is a fused protoxide of lead, and is generally obtained from the silver lead works. It oxidates nearly all the metals, excepting mercury, silver, palladium, platinum, &c., and generally forms very fusible compounds with the oxides. These two properties cause it to be a very valuable agent in separating silver and gold from all the substances with which they may be mixed.

Litharge is essentially protoxide of lead, but is occasionally mixed with a little of the red oxide; its presence is often not inconvenient, excepting it is in large quantity; it then becomes injurious, as it has the property of oxidating silver. Ordinary litharge can readily be freed from this oxide by fusing it and pouring it into a cold ingot mould, then pulverizing and carefully keeping it from contact with air, as it readily absorbs oxygen; and if it be allowed to cool in the atmosphere, it will nearly all be converted into the red oxide.

Ceruse is a carbonate of the protoxide of lead. As it does not contain the slightest traces of red oxide, it may be used where the presence of that substance may be inconvenient; but it is troublesome to use, as it is much lighter than litharge, large vessels must be employed in consequence; besides, it generally contains a small quantity of acetate or sub-acetate of lead, and about 5 per cent. of metallic lead separates from it

when it fuses, which is, in some cases, disastrous to the result of an experiment. When ceruse is employed, a certain quantity must be fused to ascertain if any metallic lead be produced;* and on the other hand, it must be examined to ascertain if it be adulterated with sulphate of baryta. When it is pure, it dissolves completely in acetic or nitric acid.†

ACTION OF OXIDE OF LEAD ON THE METALS.

The following are the results of the experiments of Berthier on the action exercised by oxide of lead on sulphur, selenium, tellurium, arsenic, and the most common of the metals. The following experiments were made in a furnace capable of producing heat enough for a copper assay.

Sulphur.—Oxide of lead is completely reduced by sulphur, with the formation of sulphurous acid, but not a trace of sulphuric acid.

Selenium is dissolved by oxide of lead in all proportions; but these bodies exercise no action on each other.

Tellurium is strongly attacked and converted into telluric acid, and combines with the oxide of lead when the latter is in excess. If the contrary be the case, the excess of acid is volatilized and telluret of lead produced.

Arsenic.—When metallic arsenic is heated with litharge, if the latter be employed in great excess, all the arsenic is oxidated; if not, a part only is oxidized,

* Berthier.

† I have never yet met with a sample which produced metallic lead on fusion.

and lead reduced, the remainder volatilizes or forms an arseniuret of lead. Mixtures of:

	1	2	3
Arsenic . .	75.24	37.60	9.40
Litharge .	111.60	111.60	111.60

gave: No. 1, a lamellar metallic button, and a compact vitreous slag of a fine orange-colour. The fusion was accompanied by a considerable arsenical smoke.

No. 2, a semi-ductile metallic button, with a lamellar fracture, like galena, but not so blue, and a transparent vitreous orange-coloured slag.

The third, a button of lead and a deep olive-green slag, very crystalline, and in large plates. This fusion was not accompanied by smoke. It is probable that arsenious acid is formed in these reactions; the last slag contained about a fifth of its weight.

Lead reduces, in part, arsenious acid; in the same manner, arsenic partly reduces oxide of lead.

Arsenious acid . . .	12.40
Lead	38.80

produced in fusing a very arsenical vapour, and produced 32 parts of arseniuret of lead, which was deep grey, semi-ductile, and had a granular fracture, a fine orange-yellow vitreous arsenite of lead was also produced.

Antimony.—The two following mixtures of antimony and litharge:

	1	2
Antimony . . .	10	10
Litharge . . .	40	80

gave (No. 1), 23 parts of lead, and a compact, well fused

slag, of a topaz yellow colour, which contained rather more than one-third of its weight of protoxide of antimony. The second gave 26 parts of lead, and a very fluid glass, which cooled rapidly, was opaque, and like yellow wax; it contained:

Oxide of lead . . .	52
Protoxide of antimony .	11.86

Tin. — This metal, cut into small fragments, was heated with the following quantities of litharge:

Tin . .	10	10	10
Litharge	37.5	80	120

The first mixture gave a slaggy substance, of dull grey colour, not well fused, with globules of lead at the lower part.

The second mixture gave 26 of lead, and a semi-fused slag, compact and opaque, the colour yellowish-grey. It contained:

Oxide of lead . . .	52
Protoxide of tin . . .	11.4

The third mixture produced 26.3 of lead, and a very fluid slag, which was compact, opaque, and greyish yellow, with a granular fracture. It contained:

Oxide of lead . . .	97.0
Oxide of tin . . .	11.4

Zinc.—10 parts of zinc filings and 100 of litharge were heated together; as soon as the latter softened, action commenced. A slight bubbling and flaming occasioned by the combustion of a portion of the zinc took place, and on increasing the heat, the mixture fused completely. The result was a button of lead,

equal to 13 parts: it was pure and ductile, and a crystalline slag, like litharge, opake and yellowish, but in small plates. This experiment proves that about one-fifth of the zinc employed is volatilized, whilst the remainder reduces the litharge. The slag contains:

Oxide of lead . . .	877
Oxide of zinc . . .	123

Bismuth.—20 of bismuth heated with 40 of litharge, gave a ductile metallic button, tin-white, and weighing 24.3, and a crystalline slag, like litharge.

Iron.—M. Berthier heated metallic iron with litharge in the following proportions:

Iron wire . . .	10	10
Litharge . . .	100	160

The first mixture gave 40 of lead, and a pasty, compact, opaque slag, of a deep metallic black-colour, and very magnetic. There was no metallic iron, but some globules of lead were present. The slag contained about:

Oxide of lead . . .	55.9
Oxide of iron . . .	13.4

The second mixture gave a button of lead, weighing 46.6, and a very fluid, compact, opaque slag, with an unequal shining lustre, deep-brown, and very magnetic. The slag contained nearly:

Oxide of lead . . .	110
Oxide of iron . . .	13.4

Copper.—The following are the results obtained with different mixtures:

Copper	15.8	15.8	15.8	15.8	15.8
Litharge	13.9	27.9	55.8	167.4	334.8

With the first mixture, a button was produced; copper-red on the exterior, grey in the interior, weighing 17 parts, and a compact, opaque, deep-red slag. It contained:

Oxide of lead . . .	10.3
Protoxide of copper . .	2.4

and the button:

Copper	13.6
Lead	3.4

The button produced by the second mixture was exteriorly copper-red, and interiorly grey spotted with red; it weighed 17.8, and the slag was compact, reddish-brown, and opaque. The slag contained:

Oxide of lead . . .	22.3
Protoxide of copper . .	3.6

and the button:

Copper	12.4
Lead	5.2

The third mixture gave a button similar to the last, weighing 18, and a compact, opaque, reddish-brown slag. It contained:

Oxide of lead . . .	49.8
Protoxide of copper . .	3.8

The button was composed of:

Copper	12.4
Lead	5.6

With the fourth mixture, a button weighing 25.6 was produced, and a slightly crystalline, reddish-brown slag, which contained:

Oxide of lead . . .	151.28
Protoxide of copper . .	10.32

and with the fifth, a grey metallic button, weighing 23.6, and a crystalline slag in large plates, like litharge, yellowish-green and reflecting red. The analysis of the button gave:

Copper	3.6
Lead	20.0

and the slag contained:

Oxide of lead . .	313.28
Protoxide of copper . .	13.72

While on this subject, it may be as well to point out the action of the oxides of copper upon lead.

The deutoxide is speedily reduced to the state of protoxide by excess of lead. If the lead be not in excess, it is totally oxidated, reducing a corresponding quantity of the deutoxide to the minimum of oxidation.

The protoxide is reduced to the metallic state by lead, but not completely, because a certain quantity of the oxide is taken up by litharge. The following mixtures have been made the subject of experiment:

Metallic lead . .	25.9	25.9	25.9	38.8	51.8
Deutoxide of copper .	19.8	14.9	9.9	9.9	9.9

All of these gave an imperfect alloy of copper and lead, and a very fusible slag composed of oxide of lead and protoxide of copper. The first produced a very small globule of copper and a very fluid slag, having a much greater tendency to run through the body of a crucible than litharge. Cooled slowly, it was reddish-

brown, opaque, and had a lamellar texture. It was composed of:

Oxide of lead . . .	27.9
Protoxide of copper . .	17.8

The second mixture produced a button of copper weighing 4.4, and a deep reddish-brown slag composed of:

Oxide of lead . . .	27.7
Protoxide of copper . .	8.7

The button gave:

Copper	4.1
Lead	0.3

The third gave a metallic button weighing 8.8, and a deep-red, opaque slag, which contained:

Oxide of lead . . .	24.89
Protoxide of copper . .	2.11

The button contained:

Copper	5.9
Lead	2.9

In the fourth and fifth mixtures, buttons weighing 21.2 and 34.8 were produced, together with slags similar to the preceding, and containing about 8 per cent. of oxide of copper.

Silicates and Borates of Lead behave as litharge, but they oxidate less rapidly. They may be prepared by fusing together 1 part of silica, or boracic acid with 1 part of litharge. The borates are more fusible than the silicates, but their use is attended with an inconvenience—they bubble very much in fusing.

Nitrates of Potash and Soda fuse at a temperature below redness without alteration, but when heated more strongly, they lose oxygen and become nitrites. If silica be present, they are further decomposed. The action of these salts is very energetic, because they have a great tendency to decompose, and because they contain a large quantity of oxygen. They are used as oxidating agents in the purification of the noble metals, for preparing black and some other fluxes. They ought always to be employed in a state of purity.

Nitrate of Lead acts much in the same way as the two last-mentioned salts. It is prepared by dissolving litharge in nitric acid, and crystallizing the solution.

Peroxide of Manganese is easily reduced to the state of protoxide by many metals, and is a very powerful oxidating agent; but is rarely employed because all its compounds are very infusible. It is employed occasionally in the purification of gold and silver. It is found in great quantity in Devonshire.

Deutoxide of Copper is not employed as a flux, but is often contained in substances submitted to assay; it then acts as an oxidating agent. A great number of metals, even silver, reduce it to the minimum of oxidation, and other metals, as iron, for instance, totally reduce it.

Peroxide of Iron.—This, like deutoxide of copper, acts sometimes accidentally as an oxidating agent.

The Caustic Alkalies are Potash and Soda. They fuse below a red heat, and then volatilize sensibly at a higher temperature; the vapours produced are abundant. Charcoal decomposes the water combined

with potash and soda, converting the hydrate into carbonate; but an excess decomposes the carbonate, and potassium is the product. Sulphur, phosphorus, &c., separate the water from their hydrates by decomposing it.

Carbonate of Potash and Soda are very much employed as agents in the assay by the dry way. They have the power of oxidating many metals, as iron, zinc, and tin, by the action of the carbonic acid they contain. A part of it is decomposed with the formation of carbonic oxide, and a compound is produced made up of alkali, carbonic acid and the metallic oxide. When fused with iron, a compact, granular, opaque body is produced, which is deep-grey and very magnetic. With zinc, a white matter is formed during the operation; a portion of the zinc volatilises and burns off. With tin, a very fluid, compact, crystalline, wax-yellow substance is the result.

The alkaline carbonates attack neither lead, antimony, or copper.

The Sulphates of Lead, Copper, and Iron.—These three salts oxidize the greater part of the metals, even silver. It is the sulphuric acid which oxidates, giving off sulphurous acid. They are used in certain processes in the assay of gold.

Sulphate of Soda is not used by itself as a re-agent, but is often a product in many operations; it is either formed in the course of an assay, or because some of the bodies employed contain it. On account of the great affinity of soda for sulphuric acid, it only oxidates those metals which combine with that element readily, as iron and zinc, for example,

DESULPHURATING RE-AGENTS.

1. The oxygen of the atmosphere.
2. Charcoal.
3. Metallic iron.
4. Litharge.
5. The caustic alkalies.
6. The alkaline carbonates.
7. Nitre.
8. Carbonate of lead.
9. Sulphate of lead.

The Oxygen of the Atmosphere acts as a desulphurating agent in roasting, when it combines with the sulphur present, forming sulphurous and sulphuric acids.

Charcoal decomposes many sulphurets by taking their sulphur to form sulphuret of carbon. It acts in this manner with the sulphurets of mercury, antimony, and zinc. Nevertheless, it is only employed as an auxiliary to the desulphurating power of the alkalies and their carbonates.

Iron separates sulphur from lead, silver, mercury, bismuth, zinc, antimony and tin, but only partially decomposes the sulphuret of copper. It is generally used in the state of filings, or nails, the latter are preferable, and ought to be kept free from rust. Oxide of iron may be used if it be mixed with the requisite quantity of charcoal to reduce it. Cast iron must not be employed, as it has very little affinity for sulphur.

Litharge exercises a very energetic action on the sulphurets, even at a low temperature. If it be employed in sufficient proportion, the sulphuret acted

on is wholly decomposed. The sulphur is often disengaged as sulphurous acid, and the metal remains alloyed with the lead proceeding from the reduction of a portion of the litharge, or combines as oxide with that portion of the litharge, which is not reduced. The quantity of litharge requisite for the decomposition of a sulphuret is considerable, and varies according to its nature; some sulphurets require 34 times their weight. When less than the requisite quantity is used, only a portion of the sulphuret is decomposed, and a corresponding quantity only of lead produced, whilst the remainder of the sulphuret forms, with the litharge and the metallic oxide which can be produced, a compound belonging to the class of oxi-sulphurets, which is generally very fusible. Oxide of lead and the sulphurets are so strongly united in this class of compounds, that galena, which is so readily attacked by pure litharge, cannot separate the least portion of lead from an oxi-sulphuret when the latter is saturated with oxygen, and it may even be introduced into the compounds without undergoing any alteration.

Many oxides, by combining with the oxide of lead, much diminish its decomposing action on the sulphurets. When litharge is heated with sulphur, its decomposing action is limited by the chemical affinity of that portion of sulphur which combines with it, and by the affinity of the metallic oxide resulting from the partial decomposition of the sulphuret. But by the addition of a suitable quantity of litharge, the sulphuret which it contains will be wholly decomposed.

When the sulphurets have a very strong base, as an alkali or alkaline earth, no sulphurous acid is given

off by the action of litharge, but all the sulphur is converted into sulphuric acid.

Litharge is a very valuable re-agent, and its use is nearly exclusively confined to the assay of the sulphurets of the noble metals; for instance, in the estimation of such metals they are obtained as alloys of lead, which are afterwards assayed by cupellation.

The following is an account of the behaviour of this re-agent with the ordinary sulphurets.

Sulphuret of Manganese requires at least six times its weight of litharge to produce a fusible compound, and thirty times its weight to desulphurize it completely. The sulphur and metal oxidize simultaneously, and a protoxide of manganese is formed, which partly peroxidizes, taking a brownish tint in contact with the atmosphere. Berthier assayed the four following mixtures:

Sulphuret of manganese . .	5	5	5	5
Litharge	20	30	100	150

The first produced an infusible, greyish-black, scoriform mass, in which small plates, having the look of galena, could be discovered. It is composed of the sulphurets and oxides of manganese and lead. Much sulphurous acid is given off during the operation.

The second fused to a soft paste, and gave 17.5 of lead, and a compact, vitreous, opaque slag, of a very deep-brown colour. The slag contained about half its weight of sulphuret of manganese.

The third fused readily, and produced 31.5 of ductile lead, and a transparent, vitreous slag, of a deep hyacinth red.

The fourth produced 33.7 of lead, exceedingly ductile, and the desulphuration was complete.

Sulphuret of Iron.—Thirty parts of litharge is sufficient to scorify protosulphuret of iron; the metal is converted merely into the protoxide.

The four following mixtures:

Protosulphuret of iron .	10	10	10	10
Litharge	60	125	250	300

gave from the first a pasty, scoriform mass, colour metallic grey, and very magnetic. It was composed of the sulphurets and protoxides of iron and lead.

The second, a very fluid metallic black slag, very magnetic, opaque and possessing great lustre, and 36 of lead.

The third, a compact, vitreous, transparent slag, of a fine resin-red, and 67 of lead.

And the last, a similar slag to the former, but containing no sulphur, and 70 of lead.

When native iron pyrites was treated with the following proportions of litharge, the results were as below indicated:

Iron pyrites . .	10	10	10	10	10	10
Litharge . .	60	125	200	300	400	500

The mixtures fused very readily with an abundant disengagement of oxygen.

The first produced only a metallic button, divisible into two parts: the lower was the largest, and was a subsulphuret of lead; the other looked like compact galena, but was magnetic; it was composed essentially of the sulphurets of iron and lead, but probably contained a small quantity of their oxides.

The second and third gave black, vitreous, opaque

slags, which stained the crucibles brown, together with lead, having a granular fracture, and a deep-grey colour; the first button weighed 35 and the second 40. Both samples of lead were contaminated with a small quantity of slag, and contained from $\frac{8}{1000}$ths to $\frac{1}{100}$th of sulphur, and a small quantity of iron.

The slags from the three last mixtures were vitreous, transparent, and a fine resin-red colour; the buttons of lead weighed 45.4, 54.8, and 86 parts. A much larger proportion of litharge does not produce more than 86 of lead, proving that 50 parts of litharge completely effect the desulphuration of iron pyrites.

Sulphuret of Copper.—The following mixtures of sulphuret of copper and litharge:

Sulphuret of copper .	10	10	10	10	10
Litharge	20	30	50	100	250

fuse very readily; giving off an abundance of sulphurous acid. The slags formed were compact, vitreous, opaque, or translucid, and more or less bright red. The copper which they contain is at the minimum of oxidation.

The three first mixtures gave metallic buttons, composed of uncombined lead and sulphuret of copper.

The fourth gave 28 of lead, with a little adhering sulphuret of copper.

And the fifth gave 38.5 of pure ductile lead, the exact quantity that ought to be produced from litharge by the transformation of the above quantity of sulphuret of copper into protoxide and sulphurous acid.

The sulphuret of copper does not combine with litharge, which is an exception to the general rule. It

requires about twenty-five times its weight of litharge to decompose it completely. When litharge is combined with a certain quantity of protoxide of copper, it has no action on the sulphuret of that metal, so that every oxide has the power of decomposing it.

The desulphuration of copper pyrites requires about 30 parts of litharge.

Copper pyrites .	10	10	10	10
Litharge . .	50	100	200	300

In the first assay, the fusion was accompanied with much ebullition, and the mass remained pasty; 6 parts of ductile lead were produced, and a matter similar to galena, but deep grey, with small facets, and a brownish-black vitreous slag.

In the second, much ebullition and swelling up took place; 35 of lead, 45 of matt, and a deep brown vitreous slag were produced.

In the third assay, 49 of lead was the result. It was covered by a thin layer of matt, and a very shining, deep brown, vitreous, translucid slag.

The last mixture fused readily, almost without ebullition, and gave 72 of lead, and a compact shining slag, of a bright grey, and without the least trace of matt; the desulphuration was complete.

Sulphuret of Antimony has a great tendency to combine with litharge, and it must be heated with at least 25 parts to effect its desulphuration. By mixing these two substances in the following proportion:

Sulphuret of antimony	10	10	10	10	10
Litharge . . .	38	60	100	140	250

the three first mixtures afforded very fluid slags, com-

pact, deep-black, and slightly metallic, and buttons of ductile lead, weighing 2, 9, and 26 parts. These slags resembled the black litharge produced at the commencement of a cupellation.

The fourth mixture gave a transparent compact slag, vitreous and shining, having a splendid hyacinth-red colour, and 50 of lead.

The last produced 57 of lead, proving the desulphuration to be complete. The antimony, in this case, exists as protoxide in the slag.

M. Fournet has observed that the sulphuret of antimony has the property of carrying sulphuret of copper, and even sulphuret of silver into the compounds formed with litharge. In one of the experiments made, a double sulphuret, composed of sulphuret of silver and sulphuret of antimony in equal parts, was fused with three times its weight of litharge, gave, firstly, a button of lead, mixed with silver; secondly, a matt-like galena; and thirdly, a black slag. This slag was analysed, and found to contain from 8 to 9 per cent. of silver.

It is probable that all the sulphurets, having a strong tendency to combine with oxide of lead, have, like sulphuret of antimony, the property of determining the scorification of a certain quantity of sulphuret of silver, like all the sulphurets, which in a state of purity, are completely decomposed by oxide of lead.

Sulphuret of Zinc must be fused with twenty-five times its weight of litharge to be decomposed. Thus:

Blende	24.08	12.08	10	10
Litharge	55.78	83.68	100	250

However strongly the first mixture was heated, it

always remained pasty; 29.2 of a greyish black-lead were produced, which contained .018 of sulphur and .008 of zinc. The button was covered by a metallic-looking black substance, intermediate between a matt and a slag; it was composed of the sulphurets and oxides of zinc and lead.

The second mixture gave 35.5 of lead and a fluid slag, which was compact, opaque, and black.

The third gave 43 of lead, and a deep grey slag.

The last produced 65 of pure lead, and a vitreous slag, of an olive-colour, and translucid on the edges.

Sulphuret of Lead.—Galena and litharge, at a heat just sufficient to fuse them, combine and form an oxi-sulphuret; but if the temperature be increased, the two bodies re-act on each other, and are mutually decomposed. If 2789 parts of litharge be employed to 1496 of lead, or 1865 of litharge, to 1000 of galena, nothing but pure lead is obtained. If more litharge be employed, a portion is not decomposed, and covers the lead. If less be employed, the galena is not completely decomposed, and the lead is covered by a matt of sub-sulphuret.

But when litharge is combined with a certain proportion of sulphurets or metallic oxides, it completely loses its oxidating power on galena, even at a white heat, so that it can be combined with this substance as with the other sulphurets, without effecting its total decomposition.

The Caustic Alkalies and their Carbonates.—All the sulphurets are decomposed by caustic alkalies, and by their carbonates also; but in the latter case, carbonaceous matter must be present. In the absence of

charcoal, there are some sulphurets, as of copper, on which they have no action. In these decompositions, alkaline sulphurets are formed, and these combine with and retain a certain quantity of the sulphuret submitted to experiment. The proportion of the sulphuret which remains in combination with the alkaline sulphurets, depends on many circumstances. It is always less when a large proportion of alkali or carbonate has been employed, as it is also when a high degree of temperature has been employed, and the presence of charcoal always much diminishes the proportion. When the radical of a sulphuret is a very volatile metal, as mercury or zinc, the decomposition may be perfect.

The reduction of that portion of alkali to the metallic state which combines with the sulphur, is brought about either by the action of a portion of the sulphur of the metallic sulphuret, when the metal is but slightly oxidizable, and then sulphuric acid is formed, which remains as a sulphate in the slag, or by the action of the metal itself when very oxidizable; the addition of charcoal always prevents the acidification of the sulphur and the oxidation of the metal; it is, then, the charcoal which reduces the alkali. When the metal of the sulphuret is readily oxidated, that portion which separates from the sulphur is completely oxidated, because it combines with the oxygen of the alkali and the carbonic acid; this occurs with the sulphuret of iron; but when the metal cannot decompose carbonic acid, a part is always obtained in the metallic state, even when no sulphuric acid is formed during the

operation: this takes place with the sulphuret of antimony.

Pearlash and native soda act more powerfully as sulphurets than the carbonate of potash obtained with nitre and charcoal, or the artificial carbonate of soda, because they always contain a part of the alkali in the caustic state.

Nitre, Saltpetre, Nitrate of Potash, has a very powerful action on the sulphurets; in fact, if not modified by the addition of some inert substance, as an alkaline carbonate or sulphate, explosion may take place, and a portion of the contents of the crucible thrown out. Where an excess of nitre is used, all the sulphur is converted into sulphuric acid, and every metal but gold and silver oxidated. When only the exact quantity of nitre is employed, that is to say, just as much as is sufficient to burn all the sulphur in the sulphurets of those metals which are not very oxidizable, as those of copper, silver and lead, the metal is obtained in a state of purity, and the whole of the sulphur converted into sulphuric acid, but with the sulphurets of the very oxidizable metals, the oxygen of the nitre is divided between the sulphur and the metal.

Nitrate of Lead possesses the combined properties of nitre and litharge. It is not much used.

Sulphate of Lead is not used as a re-agent, but is often formed in the assay of lead ores. It decomposes the sulphuret of lead by burning the sulphur. It acts on many other sulphurets in a similar manner.

SULPHURATING AGENTS.

1. Sulphur.
2. Cinnabar, or sulphuret of mercury.
3. Galena.
4. Sulphuret of antimony.
5. Iron pyrites.
6. The alkaline persulphurets.

Sulphur fuses at 226°, and at 284° is very liquid. It has very powerful affinities, combines with all the gases, excepting nitrogen, and with the greater part of the metals. That kind generally known as flour of sulphur ought to be employed; and before use, the presence or absence of earthy matters ought to be ascertained, by burning a portion.

It is principally used in the preparation of the alkaline sulphurets, and in the assay of some of the noble metals, because it does not enter into direct combination with gold, &c.

Cinnabar is decomposed by many of the metals, and it is better for use as a sulphurating agent than sulphur itself, as it is less volatile.

Galena.—Many of the metals, as iron, copper, &c., separate sulphur from lead, while some others, as silver, gold, &c., do not; so that if galena be heated with an alloy of various metals, some of which decompose it, and some do not, the former are transformed into sulphurets, and the latter combine with the metallic lead which is produced. It is often employed for this purpose. It is a common ore, and readily procured. The samples employed must contain no sulphuret of

antimony, and all the matrix must be carefully separated by sifting and washing.

Sulphuret of Antimony yields its sulphur to many of the metals, but it cannot be used in the purification of gold or silver.

Iron Pyrites is a persulphuret which loses half its sulphur at a white heat. It is much employed in metallurgical operations, but not in assaying.

The Alkaline Sulphurets can support a tolerably elevated temperature without losing sulphur, but they have a great tendency to do so, to which their sulphurating power is due. By their means every metal can be made to combine with sulphur. When an alkaline persulphuret is heated with a metal, or an oxide of a metal mixed with charcoal, a fused compound, or a mixture of the sulphuret of the metal, and an alkaline sulphuret is obtained.

When they are in combination, they are held together by very feeble affinities, and their decomposition is generally effected by the mere action of water, which dissolves the alkaline sulphuret and leaves the other perfectly pure. But with gold, molybdenum, tungstenum, antimony, &c., the compound is stable and soluble in water; and it is from this fact that the alkaline sulphurets are sometimes employed in the assay of auriferous substances.

In order to effect a sulphuration by means of the alkaline sulphurets, it is much better to use equivalent mixtures of sulphur and alkaline carbonates than to prepare them beforehand. To obtain persulphuret of potassium, 46 parts of carbonate of potash, and 54 of flour of sulphur must be employed; and for persulphuret

of sodium, 40 parts of fused carbonate of soda, and 60 parts of sulphur.

When the mixture is fused in a plain crucible, sulphate of potash, or sulphate of soda is formed, because part of the alkali is reduced to the metallic state by its affinity for the sulphur, giving up its oxygen to a portion of the sulphur, which becomes sulphuric acid; but when lined crucibles are used, the carbon combines with the oxygen of the alkali, and not a trace of sulphate is produced.

FLUXES.

Fluxes are used for the purpose of causing fusion, as their name indicates, and are employed for the following reasons:

1. To cause the fusion of a body, either difficultly fusible, or infusible by itself.

2. To fuse foreign substances mixed with a metal, in order to separate the latter by its difference of specific gravity.

3. To destroy a compound into which an oxide enters, and which prevents the oxide being reduced by charcoal. The silicate of zinc, for instance, yields no metallic zinc with charcoal, unless it be mixed with a flux capable of combining with the silica.

4. To prevent the formation of certain alloys, and consequently the combination of some metals with others, as in the case of a mixture of the oxides of manganese and iron with a suitable flux, the iron is obtained in a state of purity, whereas if no flux had been added, an alloy would have been obtained. Gold

and silver can be separated from many other metals by means of a flux.

5. To scorify some of the metals contained in the substance to be assayed, and obtain the others alloyed with a metal contained in the flux, as gold or silver with lead.

6. And lastly, a flux may be employed to obtain a single button of metal, which otherwise would be obtained in globules.

Fluxes are divided into non-metallic and metallic; and the non-metallic fluxes are:

1. Silica.
2. Lime.
3. Magnesia.
4. Alumina.
5. Silicates of lime and alumina.
6. Glass.
7. Boracic acid.
8. Borax (biborate of soda).
9. Fluor spar (fluoride of calcium).
10. Carbonate of potash.
11. Carbonate of soda.
12. Nitre (nitrate of potash).
13. Common salt (chloride of sodium).
14. Black flux and its equivalents.
15. Argol (bitartrate of potash).
16. Salt of sorrel (binoxalate of potash).
17. Soap.

The metallic fluxes are:

18. Litharge (oxide of lead), and ceruse (carbonate of lead).

19. Glass of lead (silicate of lead).
20. Sulphate of lead.
21. Deutoxide of copper.
22. Oxide of iron.

Silica is employed frequently to cause the fusion of some gangues in assays made at an elevated temperature. Silica combines with all the bases, and forms with them bodies termed silicates, which are more or less fusible.

Lime, Magnesia, and Alumina.—It is known that no simple silicate is readily fusible, so that lime, magnesia, or alumina are employed, according to circumstances, to reduce a simple silicate to such a condition that it will readily fuse in an assay furnace. Sometimes, to attain this end it is requisite to use all the above-mentioned earths.

Glass is a very useful flux in certain iron assays. The kind employed must contain no lead.

Boracic Acid.—The native boracic acid, after fusion and pulverization is to be employed whenever the use of this acid is indicated. It ought to be kept in well stopped bottles.

Boracic acid has the property of forming with silica and all the bases very fusible compounds, and is from this cause a very universal flux. Nevertheless, there is an inconvenience attached to its use; it is very volatile, so that sometimes the greater part employed in an assay sublimes before it has had time to perform its office.

Borax, Biborate of Soda is an excellent and nearly universal flux, because it has the property of forming,

like boracic acid, fusible compounds with silica and nearly all the bases, and is preferable to that acid because it is much less volatile.

It may be used at a high or a low temperature. In the first case, it is employed in the assay of gold and silver because it fuses and combines with most metallic oxides, or in obtaining a *regulus*, that is to say, to separate the metals, their arseniurets and sulphurets, from any stony matter with which they may be mixed, because this salt is neither oxidating nor desulphurating. In the second case, it is employed in the assay of iron and tin ores, as in the presence of charcoal it retains but traces of their oxides, and, indeed, much less than generally remains with the silicates.

When borax is heated it fuses in its water of crystallization, and undergoes an enormous increase of volume; at a higher temperature, it fuses and forms a transparent glass, which becomes dull on the surface by exposure to air. Only the fused vitrified borax ought to be used in assays. It must be reduced to powder, and kept in well closed vessels.

Fluor Spar, Fluoride of Calcium, is rarely employed in assays, but in certain cases is an excellent flux, as will be hereafter shewn.

Carbonate of Potash and Carbonate of Soda.—It has been already proved that they possess oxidating and desulphurating power; they will now be considered as fluxes.

They are decomposed in the dry way by silica and the silicates, with the separation of carbonic acid. The presence of charcoal much facilitates this decomposition.

They form fusible compounds with the greater part of the metallic oxides; in these combinations the oxide replaces a certain quantity of carbonic acid; but these compounds are not stable, they are decomposed by carbon, which reduces the oxide, or by water, which dissolves the alkali.

On account of their great fusibility, the alkaline carbonates can retain in suspension, without losing their fluidity, a large proportion of pulverized infusible substances, as an earth, charcoal, &c.

The alkaline carbonates ought to be deprived of their water of crystallization for assaying purposes; in fact, it would be better to fuse them before use. They must, in all cases, be kept in well stopped vessels.

They may be used indifferently, but carbonate of soda is to be preferred as it does not deliquesce.

The alkaline carbonates of commerce always contain sulphates and chlorides. In ordinary cases, this causes no inconvenience, but there are circumstances under which the presence of sulphuric acid would be injurious.

Carbonate of potash can readily be procured free from sulphate and chloride by means of nitre and charcoal, as follows: Pulverize roughly 6 parts of pure nitre, and mix them with 1 part of charcoal; then project the mixture spoonful by spoonful into a red-hot iron crucible. The projection of each spoonful is accompanied by a vivid deflagration, and carbonate of potash is found in a fused state at the bottom of the crucible; it must be pulverized, separated from excess of charcoal, and kept in a dry state for use.

Carbonate of soda may be obtained in much the

same way, substituting nitrate of soda for nitrate of potash, or by repeatedly crystallizing the carbonate of commerce.

Nitrate of Potash.—Its properties have already been pointed out. The presence of silica or silicates much assists its decomposition.

Common Salt, Chloride of Sodium, was much recommended by the older assayers, either mixed with flux, or a certain quantity placed above it, for the purpose of preserving the substance beneath from the action of the atmosphere, or to ameliorate the action of such bodies as cause much ebullition. It is very useful in lead assays. When it is used, it must be previously pounded and heated to dull redness in a crucible to prevent its decrepitation.

Black Flux and its Equivalents.—Black flux is both a reducing and fusing agent. It is a mixture of carbonate of potash and charcoal in a minute state of division. It is much employed, and very serviceable. It is prepared by mixing 2 parts of argol with 1 part of nitre, placing the mixture in an iron vessel and setting it on fire by a burning coal or red-hot rod. When the combustion is finished, the substance is pulverized and sifted whilst yet hot, and kept in well stopped jars, as it rapidly absorbs moisture from the atmosphere.

Black flux is much used in lead and copper assays; but as it boils up greatly at the commencement of the operation, the crucible must not be more than two thirds full.

It can be readily imagined that, as it fuses and reduces at the same time, the relative proportions

of alkaline, carbonate, and charcoal ought to vary according to the nature of the substance acted upon; and it is often expedient to employ the greatest possible proportion of alkali to obtain the largest yield of metal. Black flux may be obtained richer in carbon by mixing 1 part of nitre with 2½ or 3 parts of argol.

Common black flux contains 5 per cent. of charcoal. The flux prepared with 2½ of tartar or argol to 1 of nitre, contains 8 per cent., and that with 3 contains 12 per cent. of charcoal.

Black flux can be replaced by anhydrous or dry carbonate of soda mixed with some reducing agent. When charcoal is employed it must be reduced to a very fine powder; in fact, it ought to be levigated.

The three following fluxes are very useful:

Carbonate of Soda . .	94	88	816
Charcoal	6	12	184

The second is very nearly equivalent to sodium and carbonic acid, and the third to sodium and carbonic oxide; but it must be observed, that whatever precautions be taken, these mixtures never become so liquid as black flux, because the charcoal tends very much to separate and rise to the surface.

Instead of charcoal, it is preferable to use sugar or starch to make a flux equivalent to black flux with carbonate of soda; the mixture must be made most intimately.

Cream of tartar, carbonized by a semi-combustion until it is reduced to half its weight, is a very

good substitute for black flux: it contains about 10 per cent of charcoal.

Argol, Cream of Tartar, Bi-tartrate of Potash.—When bi-tartrate of potash is heated in a covered crucible, a rapid decomposition takes place, accompanied by a disengagement of inflammable gases; the substance agglomerates, but without fusing or boiling up. The residue is black, blebby, and friable, and contains 15 per cent. of carbon when produced from rough tartar or argol, and 7 per cent. from cream of tartar.

These re-agents produce the same effects as black flux, and possess more reducing power, because they contain more combustible matter; but this is an inconvenience, because the excess prevents their entering into full fusion when the substance to be assayed requires but a small proportion of a reducing agent. They can be used with success in assays requiring much carbonaceous matter.

Salt of Sorrel, Binoxalate of Potash, when heated is decomposed. It decrepitates feebly, and during its decomposition is covered with a blue flame; it at first softens, and when fully fused, is wholly converted into carbonate. When the oxalate is very pure, the resulting carbonate is perfectly white and free from charcoal; but very often it is spotted with blackish marks. It has no very great reducing power.

White, or Mottled Soap is a compound of soda with a fat acid. When heated in close vessels it fuses, boiling up considerably, and during its decomposition gives off smoke and combustible gases, and leaves a residue composed of carbonate of soda with about 5 per cent.

of charcoal. Of all reducing agents soap absorbs the greatest quantity of oxygen, and as the residue of its decomposition by heat affords but little charcoal, it has the property of forming very fluid slags. Nevertheless, it is rarely employed because certain inconveniencies outweigh its advantages. These inconveniencies are, its bubbling up and its extreme lightness. It also requires to be rasped, in order to mix it perfectly with the substances it is to decompose, and it then occupies a very large volume, and requires correspondingly large crucibles. There are nevertheless cases where it may be used with advantage by mixing it with other fluxes.

Reducing power of the various Fluxes.—By fusing each of the above-mentioned reducing fluxes with an excess of litharge, the same weight of each yielded the following quantities of lead:

Common black flux made with 2 parts of tartar	1.40
Ditto with 2½ of tartar	1.90
Ditto with 3 of tartar	3.80
Carbonate of soda 94, Charcoal 6	1.80
Carbonate of soda 88, Charcoal 12	3.60
Carbonate of soda 90, Sugar 10	1.40
Carbonate of soda 80, Sugar 20	2.80
Carbonate of soda 90, Starch 10	1.15
Carbonate of soda 80, Starch 20	2.30
Crude tartar	5.60

Cream of tartar	4.50
Ditto, ditto, carbonized	3.10
Ditto, ditto, calcined	2.20
Binoxalate of potash	.90
White soda soap	16.00

By mixing rasped soap with binoxalate of potash or carbonate of soda excellent reducing fluxes may be made.

Salt of sorrel . 85 Soap . . 15	3.25
Carbonate of soda 85 Soap . . . 15	2.40

All those fluxes containing alkaline and carbonaceous substances are reducing and desulphurating, besides acting as fluxes, properly so called; they also produce another effect which it is useful to know, viz.: they have the property of introducing a certain quantity of potassium or sodium into the reduced metal. This was first pointed out by M. Vauquelin.* He found that when oxide of antimony, bismuth, or lead was fused with an excess of tartar, the metals obtained possessed some peculiar characters, which they owed to the presence of several per cents of potassium.

Metallic fluxes: *Litharge and Ceruse.*—These bodies always act as fluxes, but at the same time often produce an alloy with the metal contained in the ore to be assayed. Ceruse produces the same fluxing effect as litharge. The former is the better flux, and is very useful in a great number of assays.

Glass of Lead, Silicate of Lead.—The silicates of lead

* Annales des Mines.

are preferable to litharge in the treatment of substances containing no silica, or which contain earths or oxides not capable of forming a compound with oxide of lead, excepting by the aid of silica. It may be made by fusing 1 part of sand with 4 parts of litharge; if required more fusible, a larger proportion of litharge must be added.

Borates of Lead.—The borates of lead are better fluxes than the silicates when the substance to be assayed contains free earths; but in order to prevent them swelling up much when fused, they must contain an excess of oxide of lead. The borate of lead containing .9056 of oxide of lead and .0944 of boracic acid, is very good. Instead of borate of lead, a mixture of fused borax and litharge may be employed; it is equally serviceable.

Sulphate of Lead is decomposed by all siliceous matters and by lime, so that when these substances are present litharge is produced, which fluxes them.

Oxide of Copper is rarely used as a flux for oxidated matters, but is sometimes employed in the assays of gold and zinc to form an alloy with those metals. In this case a reducing flux must be mixed with the oxide. Metallic copper may be used, but is not so useful, as it cannot be so intimately mixed with the assay.

Oxides of Iron are good fluxes for the silicates. They are, however, rarely employed for that purpose; they are more often used to introduce metallic iron into an alloy to collect an infusible, or nearly infusible metal, by alloying it with iron, such as manganese, tungstenum, or molybdenum.

CHAPTER IV.

ON THE BLOW-PIPE AND ITS USE—DISCRIMINATION OF MINERALS, &c.

NOTWITHSTANDING the able works already written on this portion of my subject, I should think the present deficient were I not to give a short account of the blow-pipe, its method of use, &c. I am the more inclined to do this, from the fact that the instrument to be presently considered is of much importance to the mineral analyst, (saving him, in some cases, days of needless labour); that these pages would not be that which they were intended, viz. a complete Guide to Practical Assaying, without giving short rules for its use. Having premised thus much, I hope my readers will excuse me carrying them over a path so well explored.

The blow-pipe formerly was only used by jewellers and workers of metal for producing sufficient heat for soldering certain small portions of their work; and it was only about the year 1738, that Anton Swab applied it to the analyses of mineral substances. Cronstedt used the blow-pipe to ascertain the difference between various mineral substances as to fusibility, &c. In 1765, Von Engestrom published Cronstedt's System of Mineralogy, and added to it a Treatise on the Blow-

pipe, in which he pointed out the processes of Cronstedt.

This work attracted the attention of philosophers to this valuable instrument, and its use became more general. Bergman, after Cronstedt, extended the use of the blow-pipe beyond the bounds of mineralogy to the inorganic kingdom, and in his hands this instrument became an invaluable agent for the detection of minute portions of many metallic substances. Bergman treated the greater number of the minerals known in his time with the re-agents employed by Cronstedt, described their action, and improved many of the instruments necessary for their performance. In these experiments, Bergman, whose health did not permit him to carry out such a laborious work, was assisted in his mineralogical studies by Gahn, who became particularly expert in the use of the blow-pipe. The following is a very good example of the utility of this instrument in practised hands: "Ekeberg asked Gahn his opinion of the then newly discovered mineral, the oxide of tantalum, and Gahn immediately discovered that it contained tin, although it did not amount to more than 1 per cent."

Berzelius, after Gahn, was particularly famed for his skill with the blow-pipe, and for his improvements in form of apparatus; and it is from his excellent work on this subject, that the principal portion of the descriptive part of blow-pipes, lamps, tongs, &c., is derived.

The common blow-pipe of gas fitters, jewellers, &c., is a tube of brass, tapering towards one end, and curved at that extremity, which has an opening as fine as that

made by the finest needle; it is this opening which is held against the flame of the lamp, and air is blown through it to increase the amount of heat. In all ordinary operations, the blast is required to be kept up not more than a minute, so that the quantity of moisture exhaled from the lungs produces no inconvenience by stopping up the tube. But in certain chemical operations, this is exceedingly troublesome, as a continuous blast is required, and a large quantity of water collects in consequence, generally sufficient to mar the success of an experiment. In order to obviate this, Cronstedt placed in the centre of his blow-pipe a bulb, in which the greater part of the water collected. This form was, however, inconvenient, because if the jet of the blow-pipe were at all inclined, even for an instant, the water ran from the bulb, and filled it. There have been several methods contrived to avoid this.

Bergman overcame this defect in the following manner: he fitted to the extremity of his blow-pipe a semicircular chamber, and placed the jet in the upper part of it. The best and cheapest blow-pipe, however, I have found to be that contrived by Black. The one I am in the habit of using, is made of tin-plate, japanned, and is accompanied by several platinum jets, to be hereafter spoken of. Silver is the best material for the construction of a blow-pipe; next to that, tin-plate, and lastly, brass; blow-pipes made of the latter substance soon acquire a very disagreeable odour, which they also communicate to the hands. On an emergency, a blow-pipe may be constructed with a glass tube, but owing to its frangibility and fusibility, it is not to be recommended.

Many persons have conceived that the process of

using the blow-pipe by blowing in the ordinary manner, is a very difficult matter; and some have gone even so far as to say it is injurious. Hence, various contrivances have been made to use this instrument by other means; some have employed double bellows, others bladders, and others, again, the pressure of water; but none of these methods have afforded satisfactory results, except in the hands of the contrivers, and even in some cases the results have been very problematical; and as there is in reality nothing very difficult in acquiring the art of using the blow-pipe, and as it is in my opinion not at all injurious, and moreover, as by the ordinary method more accurate results are arrived at, the matter of the various blowing machines will not be further discussed, but the necessary description of fuel now spoken of.

Any kind of flame may be used for the blow-pipe, provided it be not too small; a candle, a lamp, or gas, may be employed; Engestrom and Bergman used common candles in preference. Berzelius employs a lamp, which is certainly much preferable to a candle. I have occasionally employed the flame of coal gas, which answers very well, but is not so good as that of a lamp. Berzelius says on this subject, "Lamps have doubtless many advantages over candles, but are not so convenient in travelling, on account of the escape of oil. The oil employed ought to be the best olive or salad oil.

"The lamp which I use has the advantage of being portable, and closes in such a manner that no oil can escape. It is made of japanned tin-plate, and is about 4 inches long, and 1 inch wide, furnished at one end

with a wick-holder, capable of being completely closed by a screw, and at the other with a ring of tin-plate, which passes over the upright end of a support. It may be mentioned, that the screw-cap is furnished with a leather washer, by the aid of which it can be rendered much tighter, and the escape of oil entirely prevented."

Sometimes a spirit-lamp is employed in assays by the blow-pipe, particularly when glass tubes are employed, as in the detection of volatile substances. In these cases they are much more convenient, as an oil lamp, in the first place, blackens the tube; and secondly, does not yield a sufficiency of heat, excepting when the blow-pipe blast is employed; then the flame has more intensity than that produced by a spirit-lamp urged by the same means.

It is very difficult to give in writing a method whereby a student may acquire the practice of using the blow-pipe; that given by Faraday,* is perhaps the clearest and most concise; it is as follows: "The practice necessary, in the first place, is that of making the mouth replace the lungs for a short time, by using no other air for the blow-pipe than that contained in it." This practice is simple in itself, and easy to acquire, but as before stated, difficult to describe. Let the student first observe, that it is easy after having closed the lips to fill the mouth with air, and to retain it so, at the same time that respiration may be carried on; and if, while the mouth is in this state, a blow-pipe be introduced between the lips, it will be found that the small quantity of air which its jet allows to pass through it,

* Chemical Manipulation.

will be amply supplied for ten or fifteen seconds by the quantity contained in the mouth; and this being repeated a few times, a ready facility for using the blow-pipe, independent of the lungs, will be acquired.

This step being taken, the next is to combine this process with the ordinary one of propelling air directly from the lungs through the mouth, in such a way, that when the action of the lungs is suspended during inspiration, the blast may be continued by the action of the mouth itself, from the air contained within it. The time of fourteen or fifteen seconds, during which the mouth can supply air independently of the lungs, is far more than that required for one or even many more inspirations; and all that is required to acquire the necessary habit, is the power of opening and closing the communication between the mouth and the lungs, and between the air and the lungs, at pleasure.

The capability of closing the passages to the nostrils is very readily proved: every one possesses and uses it when he blows from the mouth, and that of closing or opening the mouth to the lungs may be acquired with equal readiness. Applying the same blow-pipe to the lips as before, use the air in the mouth to produce a current, and when it is about half expended, open the lungs to the mouth, so as to replace the air which has passed through the blow-pipe; again cut off the supply, as at first, but continue to send a current through the instrument, and when the second mouthful of air is nearly gone, renew it as before from the lungs.

To some this may be difficult; but if the preceding instructions be followed and persevered in for a short time, a continuous blast may be kept up from ten

minutes to a quarter of an hour, without any other inconvenience than the mere lassitude of the lips, caused by compressing the mouthpiece of the instrument.

After having conquered the difficulty of keeping up a continuous blast, the student must learn how to attain the maximum of heat with the least exertion to himself. The chief points to be observed, are neither to blow too fiercely or too gently; in the first case, the force of the blast would carry away heat by the quantity of cold air thrown into the flame, and in the second, a sufficient amount of heat would not be obtained, because a less amount of air would pass into the flame than that required for perfect combustion.

The highest degree of temperature is required in testing the fusibility of many bodies, as also in the reduction of certain oxides, as those of iron, tin, &c. We have yet another class of phenomena to describe, which do not essentially depend on a high temperature; these are the processes of *reduction* and *oxidation*. In order to explain and point out the best methods of effecting these two objects, it will be necessary to enter somewhat into the nature of flame; this will be done as briefly as is consistent with perspicuity. The species of flame examined will be that of a candle, as it is with a similar one to that the blow-pipe operator will have to experiment.

On careful examination, it will be found that the flame of a candle or lamp may be divided into four distinct portions; firstly, a deep blue ring at the base; this consists of the vapour of the combustible, which can hardly burn because it has not acquired a sufficient temperature; secondly, a dark cone in the centre; this

is also the vapour, but heated intensely, not, however, in a state of combustion, on account of the absence of air; thirdly, of a very brilliant envelope, which surrounds the dark part just mentioned, this is the partially consumed vapour at a very high temperature; the luminous property it possesses is due to the precipitation and subsequent ignition of particles of solid carbon; and fourthly, of an almost invisible envelope which surrounds the luminous portion; this is the substance of the combustible in full ignition, it here mingles with the atmospheric oxygen, and is consumed. The highest degree of temperature in the whole flame is to be found at the point of contact between the luminous and this part. It must be particularly borne in mind, that the inner portions of the flame have an excess of carbonaceous matters, and the outer an excess of oxygenated matters.

Having premised thus much, we will examine the nature of the flame of a candle when acted on by the blow-pipe blast, and ascertain how far it is altered, and what are the properties of its separate parts in relation to their oxidating and reducing powers. Supposing the lighted lamp or candle be ready and neatly snuffed, place the nozzle of the blow-pipe just in the edge of the flame, and about the sixteenth of an inch above the level of the wick; when things are in this state, blow gently and evenly through the blow-pipe, and a conical jet or dart of flame will be produced, which when formed in a steady atmosphere, free from accidental draughts and currents, will be found to consist of two essential parts, the inner cone, blue, small, and well defined, the outer, brownish and vague. The greatest

intensity of heat is found a little beyond the apex of the blue flame; it is there, also, *reduction* takes place. It is formed of the combustible matter mixed with air, which, however, does not burn, because it is not sufficiently heated. The outer flame is formed by the complete combustion of the combustible matter of the inner; and it is there and just beyond it, that *oxidation* takes place.

Oxidation, as before stated, takes place at the extremity of the outer flame, hence it is termed the *oxidating* flame; in it all the combustible portions are super-saturated with oxygen. In general, the further the substance to be oxidated can be placed from the extremity of the flame, the better the operation proceeds, provided always that the necessary temperature be maintained. Dull redness is the best suited to oxidation.

Reduction.—In this operation the jet of the blow-pipe must be introduced into the body of the flame, so as only to produce a small dart, and a jet having a smaller hole than that used for oxidation ought to be employed. By operating thus, a more brilliant flame than the last is produced; it is the result of a less perfect combustion, and therefore contains a large amount of carbonaceous matter, fitting it more especially for the purpose of separating oxygen from all metallic bodies.

Berzelius says, "the most important point in blow-pipe assays is the power of producing *oxidation* and *reduction* at will." Oxidation is so easy, that to do it requires only to read a description of it; but reduction requires some practice and a certain knowledge of producing various kinds of blasts. One

of the best methods of exercise in this operation is to take a small grain of tin and place it on charcoal, then direct the blow-pipe dart upon it, it will soon fuse, and if the operator has not produced a good reducing flame, it will become covered with a crust of oxide, so that it becomes a witness against him each time this happens. The nature of the flame must be altered until, by observation, the proper kind is produced at will. The longer the button of tin is kept bright, the better and more expert the operator.

AUXILIARY APPARATUS, &c.

Supports.—The support is the substance destined to hold the material to be assayed whilst under the influence of heat. From this it will be seen that a solid body must necessarily be employed; it ought also to be exceedingly refractory, so as not to give way under the excessive heat; and lastly, ought to have no chemical action on the substances placed in contact with it. Supports may be divided into combustible and incombustible; the former is charcoal, and the latter, metal, glass, and earthenware, and in some cases, certain minerals have been employed.

Well burnt wood charcoal is the best support in most cases. Berzelius remarks that light woods make the best charcoal for blow-pipe purposes, and recommends that made from the willow. Alder forms an excellent charcoal for blow-pipe experiments.

Hard woods generally give so much ash as to render them unfit, on account of the chemical action of its contents. It is generally very ferruginous. Gahn

supposed that box-wood charcoal would be best; but on trial it was found to crack very much.

It is almost needless to observe that the charcoal must be well made, because that which scintillates, smokes, or burns with flame, is worse than useless. It ought to be cut with a saw into conveniently sized pieces, and a small hole bored in them, so as to receive the substance to be assayed. I have used a very convenient charcoal support, contrived by Griffin, of Glasgow. The following is his description* of its manufacture and properties, the latter of which I most gladly corroborate: "Several of the most important experiments performed with the blow-pipe require the assistance of charcoal, upon which the object submitted to examination is supported in the flame. The charcoal employed for this purpose should be of soft wood, well burnt, compact, and free from crevices. Such charcoal is difficult to obtain. I have several times examined a sackful of charcoal without finding above half-a-dozen sticks adapted for these experiments. This circumstance induced me to seek for a substitute, and having found one which seems likely to prove serviceable, I think it possible that other persons accustomed to operate with a blow-pipe, and accustomed also to feel the want of appropriate charcoal, may be willing to learn in what manner they can efficiently replace it. For this reason I have drawn up the following notice:

"The blow-pipe experiments that require the as-

* Proceedings of the Philosophical Society of Glasgow, April, 26, 1843.

sistance of charcoal may be divided into two classes. In the first class may be named the formation of beads with microcosmic salt, the trial of fusibility *per se*, and the roasting of the metallic compounds that contain such volatile elements as sulphur and arsenic. The second class of experiments is restricted to the fusion of minerals or metallic compounds with carbonate of soda, or with soda and borax, for the purpose of effecting particular combinations, or of procuring their metals in a state of regulus. For these two classes of experiments I make use of two different composition supports; the first of which I call *Supports for Fusions*, and the second, *Supports for Reductions*.—They are alike in appearance. Each consists of two parts, an upper or combustible portion, and a base or incombustible portion. The former is the proper substitute for the ordinary charcoal, the under portion acting only as a crucible in which the combustible portion is contained. I shall first describe the composition and formation of the supports, and afterwards show the way to use them.

"The incombustible portion of both supports is made of fine pipe clay and charcoal powder, mixed in equal parts by weight with as much water, slightly thickened with rice paste, as is sufficient to form a stiff plastic mass.

"The combustible portion of the *Support for Fusions* consists of:

Charcoal in fine powder .	12 parts.
Rice flour	½ „
Water, about . . .	8 „

The rice is boiled in the water to form a paste, with which the charcoal is afterwards mixed into a mass of the consistency of dough.

"The upper part of the *Support for Reductions* consists of the following mixture:

Charcoal in fine powder .	9 parts.
Carbonate of soda, crystallized	2 „
Borax, crystallized . .	1 „
Rice flour	½ „
Water, about . .	8 „

The water is boiled, the soda and borax are dissolved in it, and the rice is then added to form a paste with which the charcoal is finally incorporated, and the whole well kneaded into a stiff mass. The mould in which these compositions are pressed to form the supports is made of boxwood.

"The principal points which require attention to ensure success in this process are to have the materials in the state of a very fine powder, and the moist compositions of a proper degree of consistency. If they are too soft, the support will not quit the mould without losing its form. If too dry, the particles of the support will not cohere. The proper state is found after a few trials. It is most convenient to begin by making the mixture too soft, and then drying it slowly, till it is found to be hard enough to work easily. The composition is rolled into balls with the fingers. The moulds should be kept clean, and the forming parts of the pestle for the charcoal composition and the ring should be oiled. The point of the pestle for the clay composition must not be oiled, because grease

prevents the adhesion of the combustible portion of clay base.

"When the support is taken from the mould it is placed in a hot plate or a sand-bath to dry, after which the rough edges are taken off by a rasp. It is then ready for use. The bottoms of *Supports for Reductions* are painted with red ochre, mixed with rice paste, to distinguish them from the other kind. The size I have fixed upon is as follows: height, half an inch diameter, at top half an inch, at bottom two fifths of an inch. The weight is about 16 grains, consisting of 10 grains of clay crucible, and 6 grains of combustible matter. I have tried several other sizes, but this I find to be the most generally convenient. Nevertheless, a higher temperature can be produced upon a smaller support, and I find that large masses of charcoal are not essential, since many blow-pipe experiments can be finished during the combustion of only two grains of charcoal. When in use, they are supported by a handle made of wire, turned into the form of a ring.

"The following is the method of using these supports:

"Firstly, the surface of one of the supports for fusion is heated before the blow-pipe till it is red hot. If then removed from the blow-pipe flame, the support continues to burn like an ordinary pastile till it is consumed down to the clay; in this respect, the support has a superiority over common charcoal, which soon ceases to burn when removed from the fire. The ignited support is then to be rested on a porcelain capsule, and a quantity of microcosmic salt, sufficient to form a

head, is placed on its red hot surface. The salt instantly melts and sinks into the central cavity, so as to form a bead, the heat, the form, and the smoothness of the surface of the support facilitating this part of the process. The salt is then heated before the blow-pipe, till it is converted into a transparent colourless bead. The support is again placed on the porcelain capsule, and the metallic substance intended to be incorporated with the bead is added to it. The support continuing to be red hot, and the bead consequently continuing soft, the substance so added is immediately absorbed, and its loss by dispersion prevented. Whereas, upon common charcoal, the fused salt solidifies soon after it is removed from the flame, and the substance added for examination not adhering to it, is often blown away by the first blast from the blow-pipe jet. The bead is now again fused, till the substance added to it is decomposed, and the resulting glass is observed to fuse quietly. It is then ready for examination, but it is sunk in the bottom of the hollow of the support, and cannot be seen by transmitted light, unless the projecting sides of the support be removed. This is effected as follows: The support is placed as before on the porcelain capsule, and the operator blows with his mouth, without the blow-pipe, strongly down upon its surface. The pastile then burns away rapidly, and the force of the blast disperses the ashes, until the whole rim of the support is consumed. The bead then appears situated on the summit of a cone, and can be examined either by reflected or transmitted light. It is also in a position adapted for exposure to the different action of the oxidating and reducing flames, so as to have the

character of the included metal fully developed. If, finally, the charcoal is allowed to burn wholly away, the coloured bead can be lifted from the ashes and preserved in a closed glass tube for subsequent examination and comparison.

"Secondly, if the surface of one of the *Supports for Reductions* be heated before the blow-pipe, it burns at first like the simple charcoal support; but in proportion as the charcoal is consumed, the fluxes which were mixed with it and which are not volatile, concentrate and fuse upon the surface of the residue. If, therefore, a reducible metallic compound is heated upon such a support, it becomes at once exposed to the reducing action of the high temperature of the nascent oxide of carbon and of the carbonate of soda, whilst any earthy matter that it may contain is vitrified by the attendant borax. For example, a crystal of sulphate of copper, as large as the surface of the support, can be decomposed upon it, and all its elements can be driven off, except the copper, which is finally obtained in a metallic bead. A globule of metallic tin, an eighth of an inch in diameter, can be kept boiling upon a support without being converted into oxide. A crystal of quartz can be fused into soda-glass. Flint-glass can be melted with metallic oxides in such quantities as to form metallic beads of enamel or coloured glass, the sixth of an inch in diameter. And these effects are producible upon a support of the weight of only 16 grains, and during the combustion, of not more than 2 or 3 grains of charcoal."

The power of restricting the consumption of charcoal

is a merit which will render these composition supports acceptable to travelling mineralogists.

Platinum.—This metal is much employed as a support in cases where charcoal would be injurious by its reducing power. It is used in three forms, viz. wire foil, and as a spoon, or small capsule.

Wire forms a support which in many cases renders the use of a foil or a spoon superfluous. It ought to be about 2½ inches long, and curved at one of its extremities, so as to form there a small circle, which is employed as a support in the following manner. After having moistened the necessary flux, place it on the curved part and fuse it in the blow-pipe dart, it will be converted into a bead, which will adhere to the curvature. The substance to be examined is then placed on the fused bead, and the whole heated together. This method of procedure is exceedingly convenient, but must not be employed where any metal is expected to be produced. This caution must be heeded in the use of all platinum vessels. To clean the wire for use after an operation, all that is necessary is to soak it in dilute hydrochloric acid, which will generally dissolve the metallic glass formed, and the wire will then be fit for a new operation. When the end of the wire has become too thin by repeated use, it must be cut off, and a new curve made. It will, by this management, be enabled to be used a very great number of times.

The platinum spoon and foil are used in much the same way; but as charcoal and the platinum wire answer every purpose, it will be unnecessary to describe their use further.

Cyanite and *Mica* have been employed as supports, but since platinum has been in use, they have been discarded.

Glass tubes.—In cases where it is necessary to roast a substance, in order to ascertain whether any volatile body is given off during that process; glass tubes, about 3 inches long, and the eighth of an inch in diameter are to be recommended. They ought to be made of hard German glass, or at all events, a hard glass containing no lead. In order to use them, the substance must be introduced, and placed at about an inch from one end; the tube is then to be so inclined that this end is the lowest; heat is then applied either by a spirit lamp or the blow-pipe flame, as circumstances require; if only a gentle heat be needed, the lamp is sufficient; if a stronger, the blow-pipe must be employed. Air may be made to pass over the ignited body with any degree of velocity, according to the inclination given to the tube; if it be horizontal, air is nearly excluded, and the more it is inclined to the perpendicular, the greater will be the current admitted. The nature of the volatile substances may be ascertained at the upper end of the tube by the smell, if gaseous; by the appearance, if merely sublimed. This is a most useful piece of apparatus, and, moreover, has great simplicity in construction and use to recommend it.

Closed glass tube, or mattrass.—This apparatus is to be employed where the presence of air is prejudicial or not necessary; as for instance, in experimenting on minerals, and to ascertain whether they contain

water or other volatile substance, or to heat such minerals as decrepitate. In cases where sulphur, for instance is required to be sublimed, the top of the tube must be closed by the finger, so as to prevent access of air, otherwise the sulphur would burn and become converted into a gaseous compound, viz. sulphurous acid.

Forceps are of several kinds, according to the use for which they are destined. There are forceps for holding fragments of mineral in the blow-pipe flame, for the purpose of ascertaining the comparative fusibility, &c.; this kind of forcep is furnished with platinum tips on one extremity, and steel nippers at the other; these latter are intended for breaking a fragment from a mineral or other hard substance.

There are also forceps for trimming the lamp or candle. These are conveniently furnished with a small spoon or shovel at the opposite end; this is used for taking up small portions of fluxes, &c.

Hammer.—This useful instrument is in constant requisition by the blow-pipe operator. Two sizes are requisite; they may be of the form recommended for those employed in the ordinary course of assay, as described in the first chapter, but must be very much lighter.

Anvil.—An anvil is also requisite to flatten grains of metal, ore, &c. It is best made of a block of steel, about 2 inches square and ½ an inch thick, polished well on both sides. It must be kept very clean.

Knife.—A knife is absolutely indispensible. It is used for ascertaining the relative degree of hardness of

minerals, for taking up portions of fluxes and pulverized ore in order to mix them together, cutting charcoal, and a multitude of other important operations. It ought to be made of the best steel, and have a stout sharp point, and a rather large handle, which enables it to be held firmly in the hand.

A few *files* are necessary. They ought to be of the best description; some may be triangular, others half round, and others round, of various degrees of fineness. They are useful for trying the hardness of minerals, cutting glass tubes, wires, &c.

Pestle and Mortar.—This piece of apparatus must be made of agate; it ought to be about ½ an inch deep, and 2 inches in diameter. When they are bought, the pestles are generally without handles, and they are in that state inconvenient in use; it is therefore adviseable to furnish them with handles, turned from some hard wood. The mortar is used for pulverizing samples of minerals, fluxes, &c., also for detecting traces of metal in any substance submitted to assay. They may be cleaned either by strong nitric acid, or friction with a piece of pomice stone; the latter method of procedure is generally the best in practice.

A series of stoppered, wide-mouthed bottles, or turned wood boxes is required, to keep the re-agents in. The bottles are preferable. A dropping tube, and a few porcelain capsules, will complete the list of apparatus, if we except a tin-plate or other box, fitted with compartments, to receive the various re-agents, &c.

Fluxes and Re-agents.—These most important bodies may be classified under two heads—the liquid and solid. They are as follows:

LIQUID.

Nitrate of cobalt.
„ baryta.
„ silver.
Nitric acid (aqua fortis).
Sulphuric acid (oil of vitriol).
Hydrochloric acid (spirits of salts).
Ammonia.
„ hydrosulphuret, (sulphide of ammonium).
„ carbonate.
„ oxalate.
Soda, phosphate.
„ carbonate.
„ sulphate.
Potash caustic.
Platinum sodio-chloride.
Alcohol.
Ferrocyanide of potassium.
Ferridcyanide of potassium.

SOLID.

Carbonate of soda.
Ammonio-phosphate of soda (microcosmic salt).
Biborate of soda (borax).
Fluor spar.
Boracic acid.
Sulphate of lime.
Pure lead.
Bisulphate of potash.
Iron wire.

Litmus paper.
Oxide of copper.
Brazil-wood paper.
Copper wire.
Tinfoil.
Nitre.
Cyanide of potassium.
Bone ashes.
Silica.

LIQUIDS.

Nitrate of Cobalt is best formed by dissolving the carbonate or oxide of cobalt in nitric acid. It ought to be free from arsenic, nickel, &c., and the solution must be moderately strong. It is used as a test for alumina, magnesia, and zinc, by the blow-pipe.

Nitrate of Baryta may be prepared by acting on the native carbonate of baryta by dilute nitric acid, until no more is taken up. The solution is then to be filtered, and evaporated to the point of crystallization. When all the crystals are formed, drain them from the mother liquor, and dry them on filtering paper. When used as a test, this salt must be dissolved in distilled water. It is employed as a test for sulphuric acid.

Nitrate of Silver.—This compound is always employed in a state of solution, and may be prepared by acting on metallic silver, by means of hot dilute nitric acid. When no more red fumes are given off, and all the silver dissolved, evaporate by a gentle heat to dryness; to expel excess of acid, redissolve in distilled water, and crystallize. When required for use, the crystals must be dissolved in distilled water. It is a most delicate test for chlorine.

Nitric Acid (aqua fortis), is best bought ready made. It is employed in the solution of various metals, alloys, and ores, and for the discrimination of certain precipitates.

Sulphuric Acid (oil of vitriol), is employed for the detection of lead, lime, baryta, and many other substances. It is in constant use.

Hydrochloric Acid (spirits of salts), is used in the solution of many minerals, precipitates, &c. It is an excellent test for silver.

Ammonia is much employed in the precipitation of metallic oxides, neutralization of acids, &c.

Ammonia, Hydrosulphuret, Sulphide of Ammonium, forms sulphurets of most of the metals when added to their solutions. Some of these sulphurets are soluble in excess of acid, some are not, and some are soluble in an excess of the re-agent itself, so that a complete separation of all the metals into three classes can be effected by means of this re-agent.

Ammonia, Carbonate is employed as a test for many of the earths; it is also employed in the removal of an excess of acid from a solution.

Oxalate of Ammonia is used as a test for lime, with which its oxalic acid forms a salt perfectly insoluble in water.

Soda, Phosphate is employed as a test for magnesia.

Soda, Carbonate.—For its uses, see *Carbonate of Ammonia.*

Soda, Sulphate is used as a test for lead, baryta, &c.

Potash, Caustic is used in the separation of iron from alumina, as also nickel from cobalt.

Platinum, Sodio-chloride is an excellent test for potash in any substance.

Alcohol is used in the detection of boracic acid, soda, &c.

Ferrocyanide and Ferridcyanide of Potassium are tests for the peroxide and the protoxide of iron; the former salt giving a blue precipitate with salts of the peroxide, and the latter, with salts of the protoxide.

SOLIDS.

Carbonate of Soda.—The plain carbonate or the bicarbonate may be indifferently employed; but in either case, it is absolutely necessary that they be free from sulphates.

There are two objects in view in the employment of soda as an auxiliary to the blow-pipe; firstly, to ascertain if the substances combining with this body be fusible or infusible; and secondly, to facilitate the reduction of certain metallic oxides.

The fusion of substances with soda.—Berzelius says, that, "Relatively to the employment of soda, there are many things to observe. The necessary quantity must be taken from its receptacle on the moistened point of a knife, and kneaded in the palm of the hand, so that it may form a coherent mass. If the body under examination be pulverulent, it must be incorporated with it but if in mass, it must be placed upon it, forcing it slightly into the moistened mass; it must then be carefully heated on the charcoal with a gentle flame, until thoroughly dry; and lastly, it may be fused. It gene rally happens that the soda, at the instant of fusion, is absorbed by the charcoal; but this does not hinder its action on the assay; for if it be fusible with soda, the latter comes to the surface and attacks it, finally form-

ing a liquid globule. If the substance be infusible in soda, but decomposable by it, it alters its appearance without entering into fusion. But, however, before pronouncing any substance to be infusible by soda, the flux ought to be mixed with the pulverized substance. If in these trials too little soda be taken, a portion of the substance remains solid, and the rest forms a covering of transparent glass; if too much, the bead of glass becomes opaque on cooling. It sometimes happens that the assay contains a substance which being insoluble in the glass of soda, prevents it becoming transparent. Then, in order that we may fall into no error respecting the nature of the glass, it becomes necessary in the two last-mentioned cases, to add a new quantity of the body under examination, and then ascertain if a limpid globule cannot be obtained. In general, it is the best method to add the soda by successive small doses, and note the changes produced by each addition. It sometimes happens, in this kind of assay, that the glass becomes coloured at the moment of cooling, and finally takes a yellow or deep hyacinth red; it even becomes occasionally opaque and yellowish brown. These phenomena indicate the presence of sulphur, either in the assay, or the soda employed. If the same colour be constantly produced by the same soda, it is a proof that it contains sulphate of soda; it must then be discarded; but if it give generally a colourless glass, it is the substance under assay that contains sulphur or sulphuric acid."

Reduction of Metallic Oxides.—This species of assay, by which quantities of reducible metals, so small as to escape the best humid analyses, can be detected, is the

most important discovery Gahn made in the application of the blow-pipe.

If a small quantity of native or artificial oxide of tin be placed on charcoal, it requires a long blast and a skilful operator to produce a grain of metallic tin; but if a small quantity of soda be added, the reduction takes place readily, and so completely with pure oxide, that the whole is transformed into a button of tin. From this it is certain, that the presence of soda favours the decomposition, but in what manner? Berzelius says that the reason is not known.

The action, however, can be explained thus: as Berzelius himself hints: "The red-hot charcoal re-acts upon the carbonate of soda, producing by its reduction a certain amount of sodium, which by its strong attraction for oxygen, seizes on that contained by the metallic oxide which is required to be reduced." If the metallic oxide contains an irreducible substance, the reduction of the former becomes difficult; but if a little borax be added, the reduction takes place as usual.

This assay is very easy of execution, and the metal is moreover readily recognized, as by previous assays the nature of it is somewhat ascertained, and the reduction but confirms the previous idea.

Supposing, however, that the metallic oxide be mixed with such a quantity of non-reducible substances that its nature cannot be ascertained by previous experiment, how can it be proved that a reducible metal is present?

Gahn has solved this question in a very simple manner. "After having pulverized the substance to

be assayed, it is kneaded in the palm of the hand with moistened soda, and the mixture placed on charcoal and exposed to a good reducing flame; a little more soda is then added, and the blast recommenced. As long as any portion of the substance remains on the charcoal, soda is added in small portions, and the blast continued until the charcoal has absorbed the whole of the mass. The first quantities of soda serve to collect the metallic particles scattered in the substance to be assayed, and the final absorption of the latter completes the reduction of any that may remain in the state of oxide.

"This done, the burning charcoal is extinguished with a few drops of water; then having cut out the part which absorbed the soda and assay, grind it to a very fine powder in an agate mortar. This powder is then washed with water to carry away the finest portion of the charcoal. The grinding and washing are repeated until all the charcoal is washed away. If the substance contained no metallic body, nothing will remain in the mortar after this last washing. But if it contained the smallest quantity of reducible matter, it is found at the bottom of the mortar, as small brilliant plates if it be malleable, or as a fine powder if it be brittle or not fusible. In either case, the bottom of the mortar is covered by metallic traces, resulting from the friction of the particles of metal against its sides, (provided that the quantity of metal contained in the sample be not too small.) The flattening of almost imperceptible globules of any malleable metal converts them into shining discs of a perceptible diameter. In this manner

may be discovered by the blow-pipe, in an assay of ordinary size, less than ½ a per cent. of tin, and even less than that of copper."

The following points in this class of assay ought to be particularly attended to. Firstly, to produce the strongest possible flame, taking care that it covers every part of the assay. Secondly, to leave none of the metal in the charcoal, or lose the smallest quantity in the collection. Thirdly, to well grind the carbonaceous mass. Fourthly, to decant very slowly, so that only the lighter parts may be carried away by the water. Fifthly, not to judge of the result until the whole of the charcoal has been removed, for a small quantity remaining suffices to hide the metallic particles; and, moreover, the particles of charcoal, viewed in a certain light, have themselves a metallic lustre, which will deceive an inexperienced eye. Sixthly and lastly, not to trust to the naked eye, however plain the sample may be, but always examine by the aid of a good microscope.

The metals reducible by this process are (besides the noble metals), molybdenum, tungstenum, antimony, tellurium, bismuth, tin, lead, copper, nickel, cobalt and iron. Amongst these, antimony, bismuth and tellurium volatilizes easily when they are exposed to a strong heat. Selenium, arsenic, cadmium, zinc, and mercury volatilize so completely, that they cannot be collected except by means of a small subliming apparatus.

The reduction can always be effected the first time when the assay contains from 8 to 10 per cent. of metal; but in proportion as the standard decreases, more attention and care must be paid to the washing

and recognition of the reduced metal in the mortar. A good system of practice in this experiment is to employ any cupreous substance, and make on it a great number of experiments, taking care to mix it each time with a substance containing no copper; thus the metallic value will diminish at each new assay, until at last no copper can be found.

If the substance to be assayed contains several metals, the reduction of their oxides must be made *in globo*, and a metallic alloy obtained. Some, small in number, are reduced separately. For instance, copper and iron give a regulus of each metal, copper and zinc: the first gives a regulus of copper, whilst the latter volatilizes. But when the result of the operation is an alloy, recourse must be had to the re-actions produced by other fluxes to ascertain its constituents.

Ammonio-phosphate of Soda, Microcosmic Salt, is obtained by dissolving 16 parts of sal ammoniac in a very small quantity of boiling water, and mixing with it 100 parts of crystallized phosphate of soda, dissolving the whole on the fire, filtering the boiling liquid, and during cooling, the double salt crystallizes. When microcosmic salt is not pure, it forms a glass, which becomes opaque by cooling. It is then necessary to dissolve it in a small quantity of water and re-crystallize it.

It may be collected in large crystals, or in a pulverulent state. The crystals are in general of a suitable size for ordinary assays. Placed on charcoal, and submitted to the blow-pipe flame, it bubbles and swells up, giving off ammonia; that which remains after this treatment is an acid phosphate of soda, which fuses

readily, and forms on cooling a transparent and colourless glass. As a re-agent, it acts principally by its free phosphoric acid, and if the salt be employed in preference to the acid, it is because it is less deliquescent, costs less, and passes readily into the charcoal. By means of microcosmic salt we then ascertain the action of free acids on any substance we may wish to assay. The excess of acid it contains combines with all bases, and forms a class of double salts, more or less fusible, which are examined as to their transparency and colour. In consequence, this flux is used more particularly in the detection of the metallic oxides, most of which impart to it very characteristic colours. This flux exercises on acids a repulsive action. Those which are volatile, sublime; and those which are fixed, remain in the mass, dividing the base with the phosphoric acid, or yielding it up entirely; in which case they are suspended in the glass without being dissolved. In this respect, microcosmic salt is a good test for silicates; for by its aid silica is liberated, and appears in the glass as a gelatinous mass.

Borax, Biborate of Soda.—The borax of commerce must be dissolved in hot water, and re-crystallized before it can be used in blow-pipe analysis. Gahn made many experiments on the fusion of borax on charcoal with soda, until both salts were absorbed; a whitish metal was produced, which appeared to proceed from the vessels in which the borax was manufactured. This never happened with borax which had been re-crystallized.

Borax may be employed either in crystals, the

requisite size for an assay, or in a pulverulent form, in which case it must be taken up on the moistened point of a knife. Some operators prefer fusing the borax before use, in order to drive off its water of crystallization, and thus avoiding the tumefaction ensuing the heating of a crystal on charcoal. This, as Berzelius observes, would be an excellent precaution, provided the borax did not regain its water of crystallization; but it recovers it to a small depth, and boils up when exposed to the blow-pipe flame, although not so much as before. As for myself, I always employ plain borax, because the tumefaction is no great inconvenience, and it is not difficult to fuse a mass so tumefied into a globule.

Borax is employed in the solution or fusion of a variety of substances. It is best to commence by acting upon a scale of the substance to be examined, because if a powder be employed, the resulting action cannot be so well ascertained. The following phenomena are to be carefully watched, for in treating any substance with borax, it must be particularly noted whether the fusion takes place rapidly or otherwise; without motion or with effervescence; if the glass resulting from the fusion is coloured; and if that colour changes in the oxidating or reducing flame; and lastly, if the colour diminishes or increases on cooling, and if under the same circumstances, it loses or retains its transparency.

Some substances possess the property of forming a limpid glass with borax, which preserves its transparency on cooling, but which, if slightly heated in the exterior, (oxidating) flame, becomes opaque and

milk white, or coloured when the flame strikes it in an unequal or intermittent manner. The alkaline earths, as yttria, glucina, zinconia; the oxides of cerium, tantalum, titanium, &c., belong to this class. In order to be certain of this result we must assure ourselves that the glass is saturated to a certain point with either of the above class of bodies. The same thing, however, does not happen with silica, alumina, the oxides of iron, manganese, &c., and the presence of silica prevents the production of this phenomenon with the earths; so that alone they present this peculiar appearance with borax; but when combined with silica, (as natural silicates, for instance), no such effect is produced. This operation has received the name of *flaming*, and any substance thus acted upon, is said to become *opaque by flaming*.

Fluor Spar, Fluoride of Calcium, and Gypsum, (Sulphate of Lime.)—These two bodies (deprived of water) are used to indicate the presence of each other. If a small piece of gypsum be placed in contact with a similar piece of fluor spar they soon liquefy at their points of contact: they then combine, and form, by fusing, a colourless and transparent bead of glass, which becomes enamel white on cooling. Fluoride of calcium is thus employed as a test for gypsum, and *vice versâ*.

Boracic Acid (*fused*) is used to ascertain the presence of phosphoric acid.

Lead is made use of in cupelling argentiferous or auriferous substances; it must be free from silver. Dumas states that the best method of obtaining lead in this desirable state is to decompose the best white-

lead by means of charcoal, as it is then impossible for it to contain any other metal.

Bisulphate of Potash is employed in the detection of nitric acid, bromine, iodine and fluorine.

Litmus Paper is made by brushing over the finest writing-paper with a strong watery infusion of litmus, and allowing it to dry out of contact of acid fumes. In this state, it is of a fine blue colour, which is changed to red on contact with an acid base; it is used to detect that class of bodies. When reddened by a slight excess of an acid, it becomes blued by an alkali, and hence can be used as a test for all those substances.

Brazil Wood Paper is prepared in the same manner as the last, substituting rasped Brazil wood for litmus in making the infusion. It is used as a test for sulphurous acid, which bleaches it, and fluorine, which renders it yellow.

Tinfoil is employed to reduce certain peroxides to the state of protoxide. When it is used, a small roll of it, about ¼ of an inch long, is plunged into the fused button, and heated strongly in the reducing flame, the desired effect is then produced.

Bone-ash is employed with lead in the cupellation of those substances containing gold or silver. The following method may be employed in preparing this substance for the purpose it is required. A small portion of very finely powdered bone-ash is taken up on the point of a knife, adding to it a small portion of soda, and making the whole into a consistent paste with a little water. A hole, of the size of the intended cupel is then scraped in a piece of charcoal, and filled with the

bone-ash paste, which is then formed into a smooth hollow by means of the small agate pestle. It may then be heated gradually by the blow-pipe until perfectly dry. The substance to be assayed, previously fused with lead, is then placed in the centre of the cupel, and heated in the exterior flame. The lead thus becomes oxidated, and the noble metal, or metals, remain on the cupel. This test is so delicate, that from a very small bead of lead (commercial) a globule of silver, visible to the naked eye, may be obtained, and, in fact, it may be demonstrated that the globule is silver by dissolving it in nitric acid on a slip of glass, and precipitating it as chloride by hydrochloric acid.

Silica has the property of forming with soda a perfectly transparent glass, which is employed in the detection of sulphuric acid.

Oxide of Copper is employed to detect the presence of hydrochloric acid and chlorine.

Copper Wire is sometimes employed in ascertaining the presence of iodine and chlorine.

Nitre, Nitrate of Potash, in long and thin crystals, is employed in hastening the oxidation of those substances which do not readily combine with oxygen in the exterior flame. It is used as follows: the point of a crystal is thrust into the fused bead; but in order to prevent the cooling of the latter, the crystal is held by a pair of pliers, so that when the bead begins to cool it may be withdrawn, the bead re-heated, and the crystal employed as before, until the desired effect is produced.

Iron Wire is employed to precipitate many metals,

and in the separation of sulphur and the fixed acids with any substance with which they may be combined. The metals which can thus be precipitated, or deprived of sulphur, are copper, lead, nickel and antimony. For instance, if a small piece of iron (harpsichord) wire be placed in a substance in fusion, and acted upon by the blow-pipe, it becomes covered with the reduced metal; the latter sometimes appears as small globules.

Iron has the property of reducing phosphorus from phosphoric acid or the phosphates, giving rise to a phosphuret of iron, which forms on fusion a white, brittle, metallic globule.

Cyanide of Potassium.—This is a most useful flux, and has only lately been introduced. MM. Haidlen and Fresenius say: "We have examined its action on many oxides, sulphurets, salts, &c., in reference to its use as a re-agent combined with the blow-pipe. We prefer, in general, a mixture of equal parts of anhydrous soda and cyanide of potassium. This mixture was employed on account of the great facility with which the pure cyanide fuses. It acts, in general, so very similarly to pure soda, that it would be superfluous to describe singly the changes which each individual body appeared to undergo when exposed to its action. We cannot, however, pass over the following especial advantages which it possesses as compared with soda. Firstly, reductions are obtained with such great facility that the least practiced operator may execute reductions which would otherwise be very difficult; for instance, the reduction of tin from either its oxide or sulphuret; and, secondly, that the fused mixture of cyanide of

potassium with soda is so easily absorbed by the charcoal, that the grains of reduced metal can always be most distinctly perceived, and may be most easily separated therefrom for further examination."

Pure cyanide of potassium may be made by heating dry ferrocyanide of potassium to whiteness in close iron vessels, and dissolving the cyanide in alcohol of 60 per cent.; but the salt in its pure state is not used as a blow-pipe re-agent; it is the mixture of cyanide of potassium with cyanate of potash, formed in the readiest manner by Liebig's process, which may be thus conducted. Eight parts of ferrocyanide of potassium are rendered anhydrous by a gentle heat, and intimately mixed with three parts of dry carbonate of potash; this mixture is thrown into a red-hot earthen crucible, and kept in fusion, with occasional stirring, until gas ceases to be evolved, and the resulting mass becomes colourless. The crucible is left at rest for a moment, and the clear salt decanted from the heavy black sediment at the bottom. The ferrocyanide and the carbonate employed ought to be perfectly free from sulphuric acid.

GENERAL ROUTINE OF BLOW-PIPE OPERATIONS.

Size of the Assay.—The morsel operated on is sufficiently large when the effect of the heat and the fluxes added can be distinctly discerned. The size of the assay-piece generally recommended, is much too large; its size ought to be about that of a mustard-seed; that of the flux added, about the size of a hemp-seed. Besides, when a large piece is employed, the experiment consumes so much more time, and requires

so much more labour than a smaller piece. It is only in reductions that a larger piece may be successfully employed, because in that case, the more metal produced, the more readily can its nature be ascertained. Having thus endeavoured to fix the size of the assay, we will now lead our readers to the operations necessary in blow-pipe analysis, and in the order in which they are to be performed.

Firstly.—The substance is heated in the closed tube, or mattrass over a spirit-lamp. It may, by this treatment, decrepitate, or give off water, or some other volatile substance.

Secondly.—It is heated gently on charcoal, by aid of the blow-pipe; and as soon as warm, withdrawn from the heat, and the odour given off ascertained; volatile acids, arsenic, selenium, or sulphur may be present. The odour thus produced by the oxidating flame must be compared with that produced by the reducing flame; if any difference, it must be carefully noted. Sulphur, selenium, &c., are best detected in the oxidating flame, and arsenic in the reducing flame.

Thirdly.—The substance is examined as regards its fusibility. If it be in grains, it is better acted upon on charcoal, notwithstanding its liability to escape on the first insufflation, when they are not very fusible. But if we can choose the form, it is better to knock off a small splinter, by means of the hammer, and hold it in the flame by the platinum-pointed pincers. A fragment with the most pointed and thinnest edges ought to be selected. By thus acting, we can always ascertain at a glance if the substance be fusible or not. Infusible substances retain their sharp points and angles, which can be ascertained immediately by means of a micro-

scope. The same points are merely rounded in bodies of difficult fusibility, and in substances of easy fusion are rendered globular.

Certain substances, and particularly some minerals, change both aspect and form when exposed to the blow-pipe flame without entering into fusion. Some swell up like borax. Some of them fuse after tumefaction; others keep in that state without fusion. Some minerals give off a sort of foam on fusing, giving rise to a sort of blebby glass, which although transparent itself, does not appear so, on account of the multitude of air-bubbles it contains.

This bubbling and tumefaction takes place in the greater part of the minerals only at that temperature at which all the water is disengaged; and these ramifications appear to proceed from a new molecular arrangement, produced by the increment of heat on the constituent parts of the substance. It cannot be said that the expansion of a particular part of the substance and its formation into gas, gives rise to this, because it most often happens in those substances which contain no such substance. The minerals which generally give these indications, are the double silicates of lime, or alkali and alumina. It sometimes disappears after a few instants, and occasionally lasts as long as the substance is kept in fusion. In the latter case, it seems that the assay takes carbonic acid from the flame, which carbonic acid is transformed by the charcoal into carbonic oxide, and it is that gas which causes the bubbles.

In the employment of fluxes, it is necessary to continue the blast for a sufficiently long time, because

some substances appear infusible at the commencement of the operation, and gradually yield to the influence of the flux, and in about two minutes, enter into full fusion. The substance is best added in small quantities, and no new dose must be introduced until the former one is perfectly acted upon; so that at last, the glass arrives at that degree of saturation that it can dissolve no more; it is at this particular point the reactions are most vivid and plain. Beads of glass, not so saturated, do not give such decided indications.

Occasionally, in operating with a flux on the reducing flame, it happens that the assay-bead re-oxidates during the cooling of the charcoal, and thus the labour of a preceding operation is lost. In order to obviate this inconvenience, the charcoal is turned over, so that the bead may fall in a yet liquid state, on some cold body, as a porcelain plate.

When the colour of the bead is so intense that it appears opaque, its transparency can be proved by holding it opposite to the flame of a lamp; the reversed image of the flame can then be seen in the bead, tinged with the colour imparted to the flux by the body under experiment. The globule may also be flattened by a pair of pliers before it cools, or it may be drawn into a thin thread. In either of the last-mentioned cases, its colour can readily be ascertained.

Minerals exposed to the exterior and interior flame, either with or without fluxes, present a variety of phenomena, which ought to be carefully noted, and which, collectively, form the result of the assay. The smallest circumstance must not be overlooked, because it may lead us to ascertain the presence of a substance not

suspected. It is always necessary, in all cases, to make two assays, and compare the separate results; because it sometimes happens, that an apparently trivial fact had been overlooked in the first series of operations, which materially conduces to the good result of the experiment. We shall now proceed to indicate the reactions produced on the various oxides of the commoner metals, in a state of purity by the blow-pipe, with and without fluxes. Their hydrates and carbonates present much the same appearances.

Potash, Soda, and Lithia cannot be distinguished with any degree of certainty by the blow-pipe; their presence is best ascertained by the wet assay; that is to say, however, with the exception of soda. Potash colours the blow-pipe flame bluish, soda, yellow, and lithia, red. These indications, however, will be more fully discussed under the head of *coloured flames.*

Baryta alone is infusible. The hydrate is fusible, but soon becomes a solid crust, on account of its losing water.

Carbonate of Baryta fuses very readily into a limpid glass, and on cooling, takes the appearance of a white enamel. On charcoal it effervesces, and becomes caustic baryta; it then behaves as above stated.

With borax, baryta fuses easily into a limpid glass, with a lively effervescence. It becomes opaque by *flaming.*

With microcosmic salt it fuses easily, with a brisk effervescence, during which the globule foams and swells; after which, it is transformed into a limpid glass.

With soda it fuses and sinks into the charcoal.

With nitrate of cobalt it produces a bead, which, when hot, is brick-red. It loses this colour by cooling.

Strontia.—Alone it presents the same phenomena as baryta; as it does also with microcosmic salt and borax.

Soda does not dissolve caustic strontia. Carbonate of strontia, mixed with its own volume of soda, fuses into a limpid glass, which becomes enamel-white on cooling.

With nitrate of cobalt, strontia becomes black, or greyish-black, and does not fuse like baryta.

Lime, alone, undergoes no alteration. Carbonate of lime becomes caustic, giving off a very strong light.

With borax it readily fuses, giving a limpid glass, which becomes opaque by flaming.

With microcosmic salt it fuses in large quantity, giving rise to a limpid glass, which preserves its transparency on cooling.

Soda scarcely acts either upon lime or its carbonate, passing into the charcoal, and leaving them unaltered upon its surface.

Acted on by *nitrate of cobalt, lime* gives a blackish mass, which is infusible.

Magnesia, alone undergoes no alteration.

With borax, behaves as with lime.

With microcosmic salt, fuses readily.

With soda, no action.

With nitrate of cobalt, after a strong heat, becomes flesh-red, which tint, however, is not well seen until after perfect cooling.

Alumina alone, does not change.

With borax, fuses slowly, and forms a diaphanous glass, which becomes opaque either by cooling or flaming.

With microcosmic salt it forms a transparent glass.

With soda it swells a little, forming an infusible compound. The excess of soda is absorbed by the charcoal.

With nitrate of cobalt, it gives a fine blue colour, by a strong blast. This colour is best observed by daylight, and is very characteristic of alumina.

Molybdic Acid.—Alone, in the open inclined tube, it fuses, giving off a white smoke, which condenses in the form of a white powder on the sides of the tube. Heated on platinum foil, it fuses and smokes. The fused portion is brown, but becomes yellowish and crystalline on cooling. In the reducing flame it becomes blue.

With borax it fuses on the platinum wire, forming in the exterior flame, a colourless and transparent glass. On charcoal, in the reducing flame, the glass becomes brown, and loses its transparency.

With microcosmic salt it fuses on the platinum wire in the exterior flame, producing a transparent glass, which while hot, is greenish, but which colour it loses on cooling. In the reducing flame, the green becomes opaque, appears black or deep blue, but by cooling becomes nearly as beautiful a green as that produced by oxide of chromium.

With soda, molybdic acid fuses on the platinum wire with effervescence, forming a limpid glass, which becomes milk-white by cooling.

Acted on by soda on charcoal, the molybdic acid is

absorbed as soon as fused; and by removing the charcoal which has absorbed it, and treating it by washing and pounding, a large quantity of metallic molybdenum may be obtained.

Tungstic Acid.—Alone it blackens, but does not fuse in the reducing flame.

With borax it fuses readily on the platinum wire, forming a colourless glass in the outer flame, which does not become opaque by flaming. In the reducing flame, the glass is yellowish when it contains only a small proportion of acid, and the colour augments in intensity by cooling, becoming perfectly yellow.

With microcosmic salt, tungstic acid dissolves, forming in the exterior flame a colourless or slightly yellowish glass. In the reducing flame it becomes a fine blue, more beautiful than that of cobalt. If the acid contains iron, the glass resumes a perfectly different appearance, becoming blood-red.

Soda dissolves tungstic acid on the platinum wire, converting it into a transparent and deep yellow glass, which crystallizes on cooling, becoming an opaque-white or yellow. If we treat tungstic acid on charcoal, with a small quantity of soda in the reducing flame, a steel-grey slag is obtained, which by washing and pounding, furnishes metallic tungsten.

Oxide of Chromium.—Alone, undergoes no change.

With borax, fuses difficultly, even in small quantity. The glass has a splendid emerald green colour, which is principally developed during cooling.

With microcosmic salt it fuses in the exterior as well as in the interior flame, furnishing a deep green glass;

and a very small quantity of oxide suffices to produce this effect.

Soda dissolves oxide of chromium on the platinum wire in the exterior flame, producing a deep orange glass, which becomes yellow on cooling. In the reducing flame it becomes opaque. It is green after cooling.

Antimony and its Oxides.—Metallic antimony fuses readily on charcoal, and when heated to redness, remains a considerable time in a state of ignition without the aid of the blow-pipe, disengaging a thick white smoke. This smoke is gradually deposited on the charcoal around the metallic globule, in small crystals, having a pearly lustre, and which, in course of time, cover it entirely. These crystals are oxide of antimony. Metallic antimony alone in the mattrass does not sublime but at the fusing point of glass. Heated to redness in the open tube, it burns slowly, giving a white smoke, which deposits on the glass, and presenting here and there traces of crystallization.

Oxide of Antimony.—Alone readily fuses, and passes off as a thick white vapour. It is reduced to the metallic state on charcoal. In this operation it colours the flame greenish.

Antimonious Acid does not fuse, but gives off a vivid light, diminishing at the same time in bulk, and covering the charcoal with a white powder, but is not reduced.

Antimonic Acid whitens at the first impingement of the flame, and is converted into antimonious acid.

The oxides and acids of antimony behave alike with fluxes.

Borax dissolves a large quantity of antimonious acid without becoming opaque. The glass continues yellow while hot, but loses nearly all its colour on cooling. When saturated, a portion of the antimony sublimes in the metallic state.

Microcosmic salt forms with the same acid a transparent and colourless glass. On the platinum wire, in the oxidating flame, it becomes yellow, which tint vanishes on cooling.

With soda, on the platinum wire, it fuses into a transparent and colourless glass, which becomes white on cooling. It is reduced on charcoal.

Oxide of Tellurium.—Alone, on the platinum wire, it fuses, giving off a smoke. It fuses and is reduced on charcoal. The reduced metal is easily confounded with antimony and bismuth.

With borax and microcosmic salt it gives on the platinum wire a limpid and colourless glass, which on charcoal becomes grey and opaque, on account of the presence of particles of reduced metal.

With soda, on the platinum wire, it produces a colourless glass, which becomes white by cooling. It is reduced on charcoal.

Oxide of Tantalum.—Alone, suffers no change.

With borax, it forms a colourless and transparent glass, which becomes opaque by flaming.

With microcosmic salt it fuses readily and in large quantity, giving rise to a colourless glass, which preserves its transparency on cooling.

Soda takes it up, and the combination is made with effervescence, but without the solution or reduction of the oxide.

As oxide of tantalum much resembles some of the earths in its re-actions with the fluxes, it may be confounded with some of them in an assay by the blow-pipe. It can be recognized from glucina, yttria and zirconia by combining with the microcosmic salt, and remaining transparent on cooling, even when the oxide of tantalum is in excess. It is distinguished from alumina by its action with borax and nitrate of cobalt; the last-mentioned re-agent having no action on oxide of tantalum, but producing a pure blue colour with alumina.

Oxide of Titanium.—Alone undergoes no change.

With borax it fuses readily on the platinum wire, forming a transparent glass, which becomes opaque by flaming. If the proportion of oxide be increased, the glass becomes opaque on cooling. Exposed to the reducing flame, the glass is yellow, if the proportion of oxide is small, becoming deep amethystine red after complete reduction, which tint becomes deeper by cooling. The glass is transparent, and very like that furnished by oxide of manganese in the oxidating flame, but is rather more blue. With a larger proportion of oxide, on charcoal, in the reducing flame, the glass becomes deep yellow, and acquires, by cooling, so deep a blue colour, that it appears black and opaque. If, however, it be *flamed*, it becomes light blue, but opaque and similar to an enamel. The tint varies in several experiments, being sometimes finer one time than another.

Microcosmic salt dissolves oxide of titanium in the outer flame, forming a colourless and limpid glass. In

the reducing flame it gives a yellowish glass while hot, but when cold it first takes a red tint, and finally becomes a very fine blueish violet. A large quantity renders the colour so opaque, that it appears black, but does not assume the appearance of an enamel. This colour may be made to disappear in the exterior flame. Its reduction is effected better on charcoal than on the platinum wire; but requires even on charcoal a sustained fire. The addition of tin much facilitates the reduction of oxide of titanium. If the oxide of titanium contain iron, or if iron be added to the glass coloured by oxide of titanium, the violet colour disappears, and the glass becomes red in the reducing flame.

Soda dissolves oxide of titanium with effervescence, forming a transparent and deep yellow glass, which does not sink into the charcoal, and becomes white by cooling. This glass possesses the property of crystallizing the instant its ignition ceases. The oxide of titanium is not reducible on charcoal, even with soda.

With solution of cobalt, oxide of titanium takes a black or greyish-black colour.

Oxide of Uranium.—Alone, blackens, but does not fuse.

With borax it fuses into a deep yellow glass, which becomes dull green in the reducing flame. The colour can be restored by exposing it to the oxidating flame on the platinum wire. On charcoal, the same operation is very difficult.

With microcosmic salt it gives a transparent yellow glass on the platinum wire, the colour of which is lessened on cooling, and finally becomes straw-yellow

with a tint of green. In the reducing flame it gives a fine green glass, which becomes yet more beautiful on cooling.

Soda does not dissolve it. With an extremely small quantity of the flux, some signs of fusion may be perceived. The mass, with a larger quantity, becomes a deep brown.

Oxides of Cerium.—Alone, the peroxide becomes protoxide, which latter does not change.

Borax dissolves the oxide in the oxidating flame, giving rise to a fine red or deep orange glass, which becomes lighter, and finally takes a yellow tint, which becomes enamel-white on flaming. In the reducing flame it loses its colour.

With microcosmic salt the oxide gives by fusion a fine red glass, which on cooling, loses its colour, taking the limpidity of water. In the reducing flame the glass becomes colourless.

With soda it does not fuse; the flux, however, passes into the charcoal.

The reactions of oxide of cerium much resemble those of iron, especially when the cerium is combined with silica. The oxides of iron and cerium do not behave in the same manner with the fluxes, excepting when they are combined with silica, as before stated, in which case it is impossible to detect cerium by means of the blow-pipe.

Oxide of Manganese.—Alone, is not fusible, but becomes brown in a strong flame.

With borax it forms a transparent glass, having the colour of amethyst, which becomes colourless in the

reducing flame. If much oxide be present, the glass must be poured on a cold body, at the instant the blast ceases. The colour returns by a slow cooling.

With microcosmic salt it fuses readily, forming a transparent glass, which is colourless in the reducing flame, and amethystine in the oxidating flame. If the glass produced by the union of oxide of manganese with phosphoric acid contain so little of the former as to give no sensible re-action, it can be rendered evident by plunging into the bead a crystal of nitre. The bead swells and foams, and the froth becomes on cooling an amethystine or pale rose tint, according to the quantity present.

With soda, the oxide fuses on platinum foil or wire, forming a transparent green glass, which becomes on cooling a blueish green. This assay is best made on platinum foil. One-thousandth of oxide of manganese gives a very perceptible colour with soda.

Oxide of Zinc.—Alone, becomes deep yellow when heated. This assay must be made by day-light. It re-assumes its white colour on cooling. It does not fuse, but gives off a vivid light during incandescence. It is gradually evaporated in the reducing flame, during the continuance of which a white ring is deposited on the charcoal.

With borax it fuses readily, and gives a transparent glass, which becomes milky by flaming, with a large proportion of oxide. It assumes an enamel-white appearance on cooling. In the reducing flame the metal sublimes, and covers the charcoal with a white film.

With microcosmic salt it behaves as with borax, except that the metal sublimes less readily with the first than the second.

Soda does not dissolve it; but acted on by this re-agent on charcoal, it is reduced, and covers the charcoal with a coating of oxide.

With solution of Cobalt it assumes a fine green colour.

Oxide of Cadmium.—Alone, in the oxidating flame, on platinum foil, undergoes no change. On charcoal it is very soon dissipated, at the same time covering the charcoal with a red or orange-yellow powder. This phenomenon is so decided in oxide of cadmium, that minerals, as carbonate of zinc, for instance, containing only two per cent. of carbonate of cadmium, on being exposed for a moment to the reducing flame, deposit, at a slight distance from the assay, a yellow or orange-yellow ring of oxide of cadmium. The cooler the charcoal is, the better it is observed. This ring forms before that of zinc, and the insufflation must not be long continued, otherwise the coloured ring will be covered by the deposit of oxide of zinc, and the operator be inclined to believe that the mineral contained no cadmium.

Borax dissolves a very large quantity on the platinum wire, forming a transparent glass, whose yellowish colour disappears on cooling. If the glass be nearly saturated, it becomes opaque by flaming, and if quite saturated, it is enamel-white on cooling. On charcoal, the cadmium is reduced, covering the former with its characteristic yellow film.

Microcosmic salt also dissolves a very large quantity,

forming a transparent glass, which, when saturated, forms a milk-white enamel on cooling.

Soda does not fuse it on the platinum wire. It is reduced on charcoal, with the production of the orange-yellow coating.

Oxide of Iron.—Alone, undergoes no change in the oxidating flame; but in the reducing flame, it blackens and becomes magnetic.

With borax it gives a dull red glass in the oxidating flame, which brightens on cooling, and finally takes a yellowish tint, or even becomes colourless on cooling. If the bead contain a very large proportion of oxide, it is opaque in the liquid state, and on cooling, becomes a dull impure yellow. In the reducing flame, it becomes bottle-green; and if the reduction be forced to the highest possible extent, assumes a lively blueish-green tint, exactly like protosulphate of iron. Tin very much accelerates the reduction of the peroxide to the state of protoxide.

With microcosmic salt it behaves as with borax, but the green colour disappears more completely, and may be entirely got rid of by the application of tin.

Soda does not dissolve oxide of iron, but causes it to be absorbed by the charcoal, in which it is easily reduced, and may be obtained as a grey, magnetic, metallic powder.

Oxide of Cobalt.—Alone, suffers no change.

With borax it readily fuses, forming a fine transparent blue glass, which does not become opaque by flaming. A very small quantity colours the glass completely blue; and a large quantity imparts so deep a colour, as to make it appear black.

With microcosmic salt the appearances are the same as with borax.

Soda dissolves but a very small quantity on the platinum wire, the fused mass is pale red by transmitted light, and becomes grey on cooling.

Carbonate of potash dissolves a larger quantity of this oxide, forming a black mass, without the slightest mixture of red. This re-action presents a method of distinguishing potash from soda.

The oxide of cobalt is very readily reduced on charcoal in the interior flame, either by an alkali or an alkaline salt. After the soda and charcoal are washed away, a grey metallic powder is obtained, which takes the metallic lustre under the burnisher.

Oxide of Nickel.—Alone, is not acted upon.

With borax it fuses very readily, producing an orange-yellow or red glass, which by cooling, becomes yellowish or nearly colourless. A larger quantity of the oxide gives a glass, which when liquid, is deep brown, but which on cooling, becomes dull red and transparent. This colour is destroyed in the reducing flame, and the glass becomes grey, on account of particles of metallic nickel being disseminated through it.

With microcosmic salt it fuses, giving rise to the same phenomena as with borax; but the colour nearly, if not quite, disappears on cooling. It behaves alike in the oxidating and reducing flames, by which reaction it is distinguished from iron. Tin produces, at first, no change; but after a short time, the nickel precipitates, and the colour disappears. The presence of cobalt, if present, can then be perceived; but the blue glass is opaque, and cannot be so well distinguished with

this flux, as when treated in the same manner with borax.

Soda does not dissolve oxide of nickel. A large quantity of this flux, however, causes the charcoal to absorb it; it is then reduced, and furnishes by washing, small white brilliant metallic particles, which are as strongly attracted by the magnet as wrought iron.

Bismuth, Oxide of Bismuth.—Alone, oxide of bismuth fuses readily on the platinum wire, forming a deep brown mass, which becomes yellow on cooling. If acted upon by a very intense flame, it is reduced, and perforates the platinum. It is reduced instantaneously on charcoal.

With borax it fuses into a colourless glass in the oxidating flame. In the reducing flame it becomes greyish, owing to the dissemination of particles of bismuth.

Microcosmic salt forms with it a brownish yellow glass. In the reducing flame, particularly with tin, a glass is formed, which is clear and colourless while hot, but becomes greyish black on cooling. The oxide of copper presents nearly the same phenomena under the same circumstance, but with this difference, that tin produces a red colour.

Owing to the facility with which bismuth may be reduced, it is nearly always on the metal that the assay is made; hence it becomes very important to distinguish it from antimony and tellurium, with which it may be readily confounded.

Firstly, in the mattrass neither antimony or bismuth sublime at a temperature the glass can bear. Tellurium, on the contrary, gives at once a little smoke,

(by means of the oxygen of the atmosphere), and finally, a grey sublimate of metallic tellurium is obtained.

Secondly, in the open tube antimony gives a white vapour, which lines the interior of the tube, and which can be driven by heat from one part to another without leaving the least trace. The metallic bead is always covered by a notable quantity of fused oxide.

Tellurium gives much vapour which attaches itself to the sides of the tube as a white powder, which is capable of fusion into colourless drops by the application of heat.

Bismuth gives no smoke if it be not combined with sulphur, and the fused metal is surrounded by the brown fused oxide, which strongly attacks the glass.

Thirdly, on charcoal these three metals give off vapour by the action of heat, and leave a ring around the spot on which they are placed. That from antimony is quite white. Those from bismuth and tellurium red or orange. If the reducing flame be made to play upon them they disappear, at the same time colouring the flame a deep green if tellurium be present, and pale blueish green if antimony. It is not coloured at all by bismuth.

Oxide of Tin.—Alone, the protoxide in the state of hydrate lights and burns like tinder, becoming peroxidized. The peroxide does not fuse or undergo any change except in the reducing flame, which if strong and long continued, entirely reduces it without the aid of any re-agent. Nevertheless, this operation requires much practice and experience.

With borax it fuses with great difficulty and in small quantity, giving rise to a transparent and colourless glass, which remains so during cooling. The colour of the glass is not changed in the reducing flame.

With microcosmic salt it behaves as with borax.

Soda and oxide of tin combine with effervescence on the platinum wire. The result of this combination is a blebby infusible mass, which cannot be dissolved by a large quantity of borax. On charcoal it is easily reduced, and gives a grain of tin.

Oxide of Lead. — Alone, minium blackens when heated, and is transformed into the yellow oxide. It forms by fusion a fine orange glass, which is reduced by effervescence on charcoal.

With borax it fuses readily on the platinum wire, and gives a transparent glass, which when saturated and hot is yellowish, but which becomes colourless on cooling. It is reduced on charcoal.

With microcosmic salt it readily fuses into a transparent and colourless glass.

With soda, oxide of lead readily fuses on the platinum wire forming a transparent glass, which becomes yellowish and opaque by cooling. Its reduction takes place instantaneously on charcoal.

Oxide of Copper.—Alone in the oxidating flame it is fused into a black bead, which is reduced on charcoal. In the reducing flame, at a temperature which does not suffice to fuse copper, the oxide is reduced, and shines with the lustre characteristic of metallic copper; but as soon as the blast ceases, the metal re-oxidates and becomes black or brown.

Exposed to a stronger heat, it gives a bead of metallic copper on fusion.

With borax, oxide of copper readily fuses in the oxidating flame, forming a beautiful green glass, which loses its colour in the reducing flame, but which on cooling becomes cinnabar red and opaque. If the oxide of copper be impure, the glass is generally deep brown, and does not become opaque but in an intermittent flame.

With microcosmic salt it fuses, attended with the same phenomena as with borax. If the quantity of copper be small, the glass occasionally becomes transparent and ruby coloured in the reducing flame; this change takes place at the instant of solidification. Commonly the glass becomes red and opaque, similar in appearance to an enamel.

When the quantity of copper is so small that the character of the red oxide cannot be made evident in the reducing flame, a small quantity of tin must be added, and the flame kept up only for an instant. The glass, previously colourless, becomes red and opaque by cooling. If the blast be kept up too long, the colour is destroyed, owing to the reduction of the copper.

With soda, on the platinum wire, a beautiful green glass is formed, which becomes opaque and colourless on cooling. On charcoal it is absorbed, and the oxide reduced. There are no means of detecting so small a quantity of copper as by the aid of the blow-pipe, that is, when it is not in combination with other metals, which by their reduction would disguise its presence. In the latter case we must use borax

and tin. When copper and iron are associated together, a single assay separates them into distinct particles; the one may be told by colour, and the other by being attracted by the magnet.

Mercury.—The compounds of mercury are all volatile, and cannot, in consequence, be distinguished by their re-action with any of the fluxes. Substances containing mercury are assayed by being mixed with a little tin, iron filings, or oxide of lead, and heating the mixture to redness in the closed tube or mattrass. In this operation, the mercury is reduced, and collects in the coldest part of the tube as a greyish powder, which being brought together by the end of a feather, collects as metallic globules. When the quantity is very small, the globules may be distinguished by aid of the microscope.

Oxide of Silver.—*Alone* is reduced instantaneously.

With borax a part is dissolved and a part reduced. In the oxidating flame, the glass becomes, on cooling, milk-white, taking the colours of the opal according to the quantity of the silver dissolved. In the reducing flame it becomes greyish, owing to the dissemination of particles of metallic silver.

With microcosmic salt the oxide and the metal give in the oxidating flame a yellowish opalline glass, seen by refraction; in the day it appears yellow, seen in the same manner, by the light of the lamp, it appears reddish.

The other noble metals, as gold, platinum, iridium, rhodium and palladium give no re-actions with the fluxes, as they are not oxidizable. They are best examined by cupellation.

Osmium is a metal found associated in the ores of platinum. It possesses the following characteristic; it is converted into an oxide which immediately volatilizes, giving a peculiar pungent odour, somewhat similar to chlorine.

Silica.—Alone undergoes no change.

With borax it fuses slowly and gives a clear glass, of difficult fusion, which is not rendered opaque by flaming.

Microcosmic salt dissolves but a very small quantity. The fused glass preserves its transparency after cooling; that which is half fused has but a semi-transparency.

With soda it fuses, giving rise to a brisk effervescence, with the production of a limpid glass.

With solution of cobalt, in certain proportions, it takes a faint blueish tint, which becomes black, or deep grey, according to the quantity of cobalt. It is by means of this colour that silica is distinguished from some aluminous substances.

Sulphur gives on burning the well known odour which is due to the formation of sulphurous acid. It leaves no residue, when pure, on being heated on the platinum foil.

COMPOUNDS OF SULPHUR WITH THE METALS, SULPHURETS.

These bodies may be recognized by the odour of sulphurous acid they exhale when heated on charcoal or in the open tube. When the quantity of sulphur contained in any compound is too small to be detected by the smell, its presence may be ascertained by fusing it with a bead composed of carbonate of soda and

silica. The glass, on cooling, takes a brown or reddish yellow colour, according to the quantity of contained sulphur. This method cannot always be employed, because the associated metals mask the colour, in which case the mineral must be roasted in the open tube, in the upper part of which is placed a piece of Brazil-wood paper. If sulphur be present, the red colour of the paper will disappear. A quantity of sulphur, so small as to be imperceptible to the smell, will bleach this test paper. This method must always be followed in the detection of sulphur in the sulphurets of antimony, because it is difficult to ascertain its presence by the smell.

The principal object, however, in view in the examination of the metallic sulphurets is to ascertain the presence of some particular metal, in which case they must be roasted, taking care to observe the precautions pointed out for the roasting of ores for assay by the furnace. The roasting must always be executed in the oxidating flame, and great care taken to apply only a very gentle heat at first, otherwise the assay will fuse, and it will then be impossible to continue the roasting with the sample. Great care must be taken to expel the whole of the sulphur, otherwise no reduced metal can be obtained by the action of soda, as sulphuret of sodium forms fusible compounds with most of the metallic sulphurets.

Selenium can be sublimed under the same circumstances as sulphur. The sublimate, if small, is reddish; but if large, so deep a colour as to appear black. It gives, when heated in the open air, according to some writers, a strong smell of decayed horse-radish,

but to me the smell most resembles that of the bisulphuret of carbon. Owing to this peculiar smell, it is very readily distinguished by the blow-pipe from all other bodies.

Seleniurets.—With the glass of silica and soda, the seleniurets behave as the sulphurets; but the colour disappears sooner by a long blast than that produced by the sulphurets. When a seleniuret is combined with a sulphuret, the selenium sublimes as selenium, while the sulphur is disengaged as sulphurous acid. If selenium be found with tellurium, the oxide of tellurium first sublimes, and, finally, the selenium is deposited nearest the point heated. Sometimes the sulphuret of arsenic sublimes with the same appearances as selenium, but never with the same odour.

Phosphorus entirely burns away, giving off a white fume which is phosphoric acid.

SALINE SUBSTANCES, SULPHATES, PHOSPHATES, IODIDES, BROMIDES, &c.

Sulphates.—The presence of this class of bodies is ascertained in the same manner as sulphur, by means of the glass of soda and silica. The sulphates of the metals proper, when heated with charcoal in the close tube, give off sulphurous acid, which may be detected either by the smell or by its action on Brazil-wood paper. The metals of the alkalies and alkaline earths give no sulphurous acid when treated in this manner.

Nitrates. — All the salts of nitric acid deflagrate with carbonaceous matters. This, however, is not

characteristic, for the chlorates also possess this property. If any nitrate be heated in the close tube with bisulphate of potash red fumes of nitrous acid are evolved.

Bromides, heated with bisulphate of potash in the closed tube, give off vapours of bromine, which are similar in appearance to those of nitrous acid, but which recal the smell of chlorine. Under the head of *Coloured Flames*, another method of distinguishing bromine will be pointed out.

Iodides, acted on by bisulphate of potash, give rise to splendid violet-coloured vapours, which are characteristic.—(*Also, see Coloured Flames*).

Chlorides, treated with bisulphate of potash and peroxide of manganese, evolve chlorine, which may be recognised by its peculiar odour and yellowish-green colour.—(*For further information, see Coloured Flames*).

Fluorides, heated with bisulphate of potash, give rise to fluoric acid, which may be distinguished by its power of corroding glass. As fluorine occurs in very small quantities in certain minerals, and as it is rather difficult of detection, full instructions will be given.

In case the mineral is very rich in fluoric acid, it may be mixed with microcosmic salt (previously fused), and heated at the extremity of an open tube, so that part of the current of air feeding the flame, can pass into the tube. Aqueous fluoric acid is then formed, which can be recognised by its odour and by the corrosive action it exercises on the tube. If a slip of Brazil-wood paper be held at the opening of the tube, it becomes immediately yellow. On the contrary, when the acid exists but in minute quantity, as in fossils, or where it is com-

bined with weak bases, or with a certain proportion of water, the substance can be heated in the close tube, after the introduction of a piece of moistened Brazil-wood paper. Hydro-fluosilicic acid is liberated by the heat, and a dull ring of silica deposited on the glass, a little above the assay; and lastly, the end of the Brazil-wood paper is turned yellow. Three or four per cent. of fluoric acid can be detected in this manner.

Phosphates.—The following is the method recommended by Berzelius for the detection of phosphoric acid. "The substance to be assayed is dissolved in boracic acid, and when a good fusion is effected, a piece of fine steel wire, a little larger than the diameter of the bead is forced into it, and the whole then exposed to a good reducing flame. The iron is oxidated at the expense of the phosphoric acid, causing the formation of a borate of the oxide of iron and phosphuret of iron, which fuses at a sufficiently high temperature. The bead is then taken from the charcoal, enveloped in a piece of paper, and struck lightly with a hammer, by which means the phosphuret of iron is separated from the surrounding flux. It exists as a metallic-looking button, attractable by the magnet, frangible on the anvil, the fracture having the colour of iron. If the substance under assay contained no phosphoric acid, the iron wire will keep its form and metallic lustre, excepting at the ends, where it will be oxidated and burnt. The substance to be assayed ought not to contain sulphuric acid, arsenic acid, or any metallic oxides reducible by iron."

Hydrates.—The presence of water in these substances can be ascertained by heating them in the close tube.

If any water be present, it will vaporize and condense on the coolest portions of the tube.

Silicates. — These compounds of silica with bases, are decomposed by fusion with microcosmic salt, the silica being set at liberty, and the base combining with the phosphoric acid. When but a small quantity of microcosmic salt is employed, it often happens that the silica swells at the moment of decomposition, absorbing the liquefied mass. By adding a large quantity of the flux, the whole can be converted into a globule, which retains in suspension the semi-transparent tumefied silica. This can best be perceived when the glass is in a state of ignition.

COLOURED FLAMES.

There are a great number of substances best detected by the colours they impart to the flame of the blow-pipe. Indeed, so important is this point, that it has been thought advisable to collect all the facts known on this subject in one place, rather than scatter them over the work. These experiments are best made in a dark room, and with a very small flame.*

BLUE FLAMES.

Large intense blue . . .	Chloride of copper.
Pale clear blue . . .	Lead.
Light blue	Arsenic.
Blue	Selenium.
Greenish blue	Antimony.
Blue mixed with green . .	Bromide of copper.

* Griffin's Blow-pipe Analysis, page 148.

GREEN FLAMES.

Very dark green, feeble	Ammonia.
Dark green	Boracic acid.
Dark green	Iron wire.
Full green	Tellurium.
Full green	Copper.
Intense emerald green	Iodide of copper.
Emerald green, mixed with blue	Bromide of copper.
Pale green	Phosphoric acid.
Very pale apple green	Barytes.
Intense whitish green	Zinc.

YELLOW FLAMES.

Intense greenish yellow	Soda.
Feeble brownish yellow	Water.

RED FLAMES.

Intense crimson	Strontian.
Reddish purple	Lithia.
Reddish purple	Lime.
Violet	Potash.

Chlorine, combined with copper, gives an intense blue flame. This phenomenon may be produced as follows: Take a piece of thin brass wire, and bend one end of it several times upon itself; place upon this some microcosmic salt, and fuse it until it has acquired a green colour. Then add to it the substance suspected to contain chlorine, and place it in the oxidating flame just at the point of the blue flame, when, if any chloride be present, a splendid blue colour will be produced.

Lead.—The blue colour produced by this metal is readily obtained. Fragments of a mineral must be held in the tongs, and powder may be assayed on charcoal.

Arsenic, in the metallic state, gives rise to a light blue flame.

Selenium and Antimony, when treated in the same manner, afford characteristic flames.

Bromine.—If any substance containing bromine be placed in a bead of fused microcosmic salt on the brass wire, and then in the oxidating flame, a bright blue flame, with emerald green edges, will be produced.

Boracic Acid.—The following is Dr. Turner's process for the detection of boracic acid. "The substance is to be mixed with a flux composed of 1 part of fluor spar, and 4½ parts of bisulphate of potash. This mixture is to be made to adhere to the moistened end of a platinum wire, and held at the point of the blue flame; at the instant of fusion, a dark green flame will be produced. It may also be produced by merely dipping the mineral in sulphuric acid, and exposing it to the blow-pipe blast. In case a very small quantity of boracic acid is contained in a mineral, the following process may be employed. The substance must be fused with carbonate of potash on charcoal, moistened with a drop or two of sulphuric acid, and then a few drops of alcohol; the latter will burn with a green flame when exposed to the flame of the blow-pipe.

Tellurium.—The peculiar flame given by this metal is produced by heating a portion of its oxide on charcoal in the reducing flame.

Copper.—All the compounds of copper, except those

in which bromine and chlorine enter, give a beautiful green flame. The soluble salts give it *per se*, but the insoluble require moistening with sulphuric acid.

Iodine and Copper.—To the bead of microcosmic salt on the brass wire, add any compound containing iodine, and a bright green flame will be produced when the mass is heated in the oxidating flame.

Phosphoric Acid.—The phosphates, when moistened with sulphuric acid, give a light green tint to the outer flame.

Baryta.—The soluble salts of baryta give a light green colour to the outer flame when moistened with water.

Zinc, when exposed to the blow-pipe flame, burns with an intense whitish-green light.

Soda.—Any salt of soda, or substance containing soda, being exposed to the outer flame, gives a brush of intensely coloured flame, of a fine amber or greenish-yellow.

Water.—Certain minerals containing water, give a feeble yellowish tint to the flame.

Strontian.—All the salts of this substance which are soluble in water, give a crimson tint to the flame, which does not endure after the substance is fused. Carbonate of strontian must be moistened with hydrochloric acid, and sulphate of strontian must be reduced to the state of sulphuret by ignition with charcoal; it must then be moistened with hydrochloric acid; after which treatment, it will exhibit the characteristic flame.

Lithia.—All that has been said of strontia applies to lithia, with the remarkable exception, that the coloured

flame given by lithia is durable, whilst that afforded by strontia is evanescent.

Lime acts as strontia.

Potash, treated as soda, gives a purplish light; but the re-actions of potash and soda with oxide of cobalt are the best tests of their presence, combined with the peculiar light afforded by soda.

ON THE INDICATIONS GIVEN BY THE MOST COMMON OF THE MINERALS ON BEING TREATED BY THE BLOW-PIPE, AIDED BY FLUXES.

MINERALS OF COPPER.

Sulphuret of Copper.—Alone, on charcoal, gives off sulphurous acid, fusing readily in the outer flame. In the inner flame it is covered with a crust, and does not fuse.

In the *open tube* sulphurous acid is disengaged, but no sublimate is produced. The residue, treated with soda and borax, gives a button of copper.

Argentiferous Sulphuret of Copper.—Alone fuses easily, giving off sulphurous acid. Cupelled with lead, on bone-ash, it leaves a large bead of silver, and the cupel appears a blackish green.

Sulphuret of Antimony and Copper, Bournonite.—Alone, in the open tube, gives off the antimonial smoke, with an odour of sulphurous acid. A slip of Brazil-wood paper, on being placed within the tube, is bleached.

On charcoal, a deposit of antimony, but no trace of lead. The bead diminishes in size, becoming grey,

and semi-malleable. Fused with soda, it gives a grain of copper.

Copper Pyrites, Sulphuret of Iron and Copper.—Alone, on being heated, blackens, becomes red by cooling, and fuses more easily than the sulphuret of copper, finally giving a bead attractable by the magnet. This bead is brittle, and reddish-grey in the fracture. If after a long exposure to the oxidating flame, it be treated with a small quantity of borax, a regulus of copper is obtained.

In the *open tube,* much sulphurous acid is given off.

With soda, globules of iron and globules of copper are obtained, provided the ore has been sufficiently roasted.

Sulphuret of Tin and Copper, Tin Pyrites.—Before the blow-pipe it becomes, by roasting, covered with a snow-white powder, which is oxide of tin. The white powder also encircles the globule to the extent of about two lines.

In the *open tube,* sulphurous acid is given off.

Needle-ore, Aikenite.—Alone, it fuses, giving off vapour, which coats the charcoal snow-white, slightly yellowish on the interior edge, finally giving a metallic bead resembling bismuth.

In the *open tube* it gives off a white smoke, one part of which is fusible, and the other volatile. The first part is converted by fusion into limpid drops, which become white by cooling; there is also an odour of sulphurous acid. Treated by fluxes, the resulting bead of bismuth gives the re-action of copper. After a long blast, a grain of copper may be obtained, which by cupellation with lead gives traces of silver. The fusible white

smoke, at the commencement of the operation, indicates the presence of tellurium.

The Oxides of Copper.—The action of the fluxes, &c., on these bodies has already been pointed out.

Chloride of Copper.—Alone, colours the flame blue, with greenish edges. A red pulverulent deposit forms on the charcoal around the assay; the fused matter reduces, giving a grain of copper, surrounded by slag.

With the fluxes, the chloride behaves as the oxides.

Carbonate of Copper.—Alone, in the mattrass, gives water, and blackens.

On charcoal, it fuses, and behaves like oxide of copper.

Arseniate of Copper behaves with the fluxes in the same manner as the oxide of copper, but exhales a strong odour of arsenic, and gives, when reduced with soda, a white and brittle bead.

ORES OF LEAD.

Sulphuret of Lead, Galena.—Alone, on charcoal, does not fuse until after disengagement of sulphur; globules of lead then form on the surface, and finally, a bead of lead is obtained. By cupelling thus, the presence of silver may be ascertained. After cupellation, the bone-ash indicates by its colour whether the lead were pure or not; if it were, when cold the cupel would be pure yellow; copper renders it green and iron-brown, or blackish.

In the tube, galena gives off sulphur, and a white sublimate of sulphate of lead.

Oxide of Lead.—Its action with fluxes has been already shown.

Sulphate of Lead decrepitates, fuses on charcoal, in the outer flame into a transparent bead, which becomes milky by cooling. In the reducing flame it effervesces, giving a button of lead.

Carbonate of Lead behaves like oxide of lead.

Phosphate of Lead.—Alone, on charcoal, it fuses, the bead crystallizing as it cools. The crystals have large facets, and a pearly whiteness.

With the fluxes it behaves like oxide of lead.

ORES OF ZINC.

Zinc Blende, Black Jack, Sulphuret of Zinc.—Alone, decrepitates violently. Suffers no remarkable change on ignition; does not fuse, and gives off but a very slight odour of sulphurous acid, being very difficult to roast.

On charcoal, an annular deposit of oxide of zinc is formed when heated violently in the outer flame.

Soda attacks it feebly; but the zinc is reduced in a good fire, with the deposition of oxide of zinc on the charcoal.

Carbonate of Zinc, Calamine.—Alone gives off no water, but becomes a white enamel, which behaves like oxide of zinc.

ORES OF TIN.

Oxide of Tin.—Its behaviour with fluxes has already been noticed.

ORES OF IRON.

Sulphuret of Iron, (magnetic pyrites).—Alone, undergoes no change.

In the *open tube,* gives sulphurous acid.

On charcoal, becomes red in the outer flame, and is changed, by roasting, into an oxide of iron.

Common Pyrites.—Alone in the mattrass, gives sulphuretted hydrogen and sulphur.

On charcoal it behaves like magnetic pyrites.

Mispickel, Arsenical pyrites.—Alone, gives first a red sublimate, which is sulphuret of arsenic, then a black; and lastly, in a strong fire, metallic arsenic sublimes. Treated on charcoal, the residue gives no arsenical odour, and behaves like magnetic pyrites.

On charcoal, mispickel gives a thick smoke of arsenic, then fusing, exhaling the odour of that metal. If the mispickel contain cobalt, it can be detected after well roasting the ore, and fusing the residue with borax or microcosmic salt; and after cooling, the glass takes the characteristic colour of cobalt.

Magnetic Oxide of Iron, and Oxide of Iron, behave as already described.

Carbonate of Oxide of Iron, heated in the mattrass, gives no water. Some species decrepitate strongly. Exposed to a gentle heat, it blackens, and gives oxide of iron, very attractable by the magnet.

Chromate of Iron.—Alone, undergoes no alteration. With borax and microcosmic salt, the solution is slow but complete. The characteristic colours are alone apparent when the bead is hot; but as soon as it cools,

the fine green of chromium makes its appearance. This re-action is most intense when the substance is treated in the reducing flame, and appears in all its lustre by the addition of tin.

Hydrate of Iron gives water in the mattrass, and leaves red oxide after fusion with microcosmic salt; it gives with tin some traces of copper.

ORES OF MANGANESE.

Oxides of Manganese.—The characteristic reactions of this class of bodies have already been discussed.

COBALT ORES.

Sulphuret of Cobalt, in the mattrass, gives no volatile substance, and does not decrepitate. In the open tube gives sulphurous acid, and a white sublimate, which consists of drops, perceptible by the microscope, they are concentrated sulphuric acid. There are no traces of arsenic.

With the fluxes, the re-actions of cobalt so predominate, that it is impossible to discover those of iron and copper; but if it be fused many times with borax, in the exterior flame, (that is, the grey bead produced by fusion on charcoal of the mineral itself), the borax takes away the cobalt, and the copper concentrates; so that when the mass is fused with microcosmic salt, and exposed to the reducing flame, the red colour of the oxide of copper is produced, shaded, however, by the cobalt blue.

Arsenical Cobalt.—Alone, in the open tube, gives an abundance of arsenious acid, with great facility. In the

mattrass, some species give a little metallic arsenic, others give none.

On charcoal, all disengage an arsenical smoke and odour, and give by fusion a white metallic bead.

Cobalt glanæ, (Tunaberg).—*Alone*, in the mattrass, suffers no change.

In the *open tube*, roasts with difficulty, giving no arsenious acid but by a very strong fire, but disengaging sulphurous acid.

On charcoal, gives an abundance of fumes, and enters into fusion after some considerable roasting; it then behaves as arsenical cobalt.

Black Oxide of Cobalt.—Alone, gives a little empyreumatic water.

On charcoal, gives traces of arsenic, but does not fuse.

Dissolves in borax and microcosmic salt, giving so deep a blue as to disguise all other action.

It is infusible with soda, and giving on the platinum wire a mass strongly tinted green by manganese.

Arseniate of Cobalt.—Alone, in the mattrass, gives off water and becomes brown, but gives no sublimate.

On charcoal, gives off much vapour, and a smell of arsenic. Fuses in a good reducing flame, and is converted into arsenical cobalt.

ORES OF MANGANESE.

Sulphuret of Manganese.—Alone, in the mattrass, undergoes no change.

In the *open tube* roasts slowly, but gives no sublimate. The roasted surface takes a bright green tinge.

On charcoal, after complete roasting, behaves with the fluxes like pure oxide of manganese.

Peroxide of Manganese.—Alone, in the mattrass, when pure, undergoes no sensible alteration, but in general, it contains more or less hydrate of manganese, the water of which may be driven off by means of heat. The more water the heated matter gives off, the less available oxide of manganese it contains. On charcoal it becomes reddish brown, in a good reducing flame.

With borax and microcosmic salt, it dissolves, with a brisk effervescence, produced by disengagement of oxygen; it then behaves as oxide of manganese.

ORES OF CHROMIUM.

Chrome Ochre.—Alone, decolorises, and becomes nearly white, but does not fuse.

Borax separates oxide of chromium, and takes a fine green colour.

It dissolves with great difficulty in microcosmic salt, and the green colour is not so beautiful as with borax.

ORES OF ANTIMONY.

Red and Black Sulphurets of Antimony.—Alone, they fuse readily on charcoal, which absorbs them and becomes covered with a black vitreous crust. After the blast has been continued for a few moments, metallic globules appear on the charcoal, which seem to be a sub-sulphuret, as they do not behave like the pure metal; for they do not burn, but blacken, and become dull on the surface after cooling.

Roasted in the glass tube, much antimonious acid forms at the commencement; that which sublimes after-

wards, is a mixture of antimonious acid with much oxide.

GOLD.

Graphic Gold.—On charcoal, fuses into a dull grey metallic bead, covering the charcoal with a white smoke, which disappears with a green or blueish light, when the reducing flame is thrown upon it. After a continued blast, a bright yellow metallic grain is obtained. It is, after cooling, brilliant and malleable.

In the *open tube* it deposits a smoke, which is white, excepting in the neighbourhood of the assay, where it is greyish. This is sublimed tellurium. This deposit forms limpid drops when the flame is directed upon it.

Telluriferous and Plumbiferous Gold.—Alone, on charcoal, it fuses like the preceding, and forms a pulverulent deposit on the support; but this deposit is yellow; it disappears in the reducing flame with a blue colour, which is not at all green. It gives, after a strong blast, a grain of gold, which ignites at the instant of congelation. This grain is malleable.

In the *tube* it fumes, giving a very sensible fume of sulphurous acid. It then gives a sublimate, which is grey close to the assay, but white elsewhere.

ORES OF MERCURY.

Cinnabar, Sulphuret of Mercury.—Alone, on charcoal, it volatilizes without residue, giving off an odour of sulphurous acid. In the mattrass, it sublimes, giving a blackish sublimate. In the open tube, it gives, by roasting, mercury and sublimed cinnabar. In the mattrass, with soda, globules of mercury are obtained.

Muriate of Mercury, Horn Mercury.—On charcoal,

volatilizes without residue. In the mattrass, gives a white sublimate. With soda, in the mattrass, gives much mercury in globules.

With microcosmic salt, fused on the brass wire, it communicates a fine azure blue colour to the flame, indicative of chlorine.

ORES OF SILVER.

Sulphuret of Silver.—Alone, on charcoal, fuses and swells considerably, forming large bubbles; but after a continued blast, it forms a grain. It gives off an odour of sulphurous acid, and finally furnishes a grain of silver, surrounded by slag. Fused with borax and microcosmic salt, the slag gives traces of copper.

Red Silver.—Alone, on charcoal, decrepitates a little, fuses, burns, and smokes, like antimony, but gives no arsenical odour. The production of vapour lasts but for a few minutes.

In the *open tube*, it gives much vapour, and a smell of sulphurous acid, which is very strong at the commencement. The deposit on the sides of the tube is sometimes crystalline; it is oxide of antimony. The bead, which remains after a long exposure to the exterior flame, is a button of pure silver.

Antimonial Silver, and Argentiferous Antimony.—Alone, on charcoal, fuses readily, forming a metallic bead, which is not malleable, giving off a vapour like that of pure antimony, but less abundant. The bead becomes, after the disengagement of a certain quantity of antimony, dull white, and very crystalline, entering into ignition at the instant of congelation. When it has lost yet more antimony, its surface becomes smooth, like glass; and the heat which it then disengages, is more intense

than at any other time. Lastly, after a long continued blast, nothing but pure silver remains.

In the tube, much oxide of antimony is given off, and the bead which remains is surrounded by a bead of deep yellow glass.

Electrum gives by fusion a grain of gold, which varies in whiteness, and which gives with borax and microcosmic salt the same reactions as pure silver.

Amalgam, in the mattrass, swells up, and gives much mercury, leaving silver, which may be fused to a bead on charcoal.

Muriate of Silver, Horn Silver.—On charcoal becomes a bead, which, according to the purity of the salt, is grey, brownish, or black. In the reducing flame it is gradually converted into metallic silver. It gives with microcosmic salt, fused on the platinum wire, a blue flame, like the muriate of mercury.

BISMUTH.

Native Bismuth.—Alone, fuses, giving a weak arsenical odour. Otherwise, it presents the same phenomena as pure bismuth.

In the open tube it gives a little arsenious acid. Cupelled, it tinges the bone-ash pure orange-yellow.

Sulphuret of Bismuth.—Alone, in the tube, gives sulphurous acid, and a white sublimate; heated to redness, it deposits oxide of bismuth round the assay, like pure bismuth.

On charcoal, it fuses with bubbling, throwing out small incandescent globules. This agitation lasts but a short time.

Oxides of Bismuth, see pages 156—157.

ORES OF NICKEL.

Sulphuret of Nickel.—In the open tube gives sulphurous acid, becomes black, but does not change form. On charcoal, gives, by aid of a good flame, a mass conglomerated by semi fusion. It is metallic, malleable, and magnetic, and is pure nickel.

After roasting in the open air, it behaves with fluxes like oxide of nickel.

Arsenical Nickel, in the mattrass gives nothing volatile; semi-fuses at the temperature which softens glass, and a deposit of arsenious acid is formed on the sides of the mattrass; this is owing to the included air.

It fuses on charcoal, with a vapour and arsenical odour, and a white metallic globule.

In the open tube it roasts easily, with the formation of a large quantity of arsenious acid; the residue is a yellowish-green substance, which on roasting afresh on charcoal, and fusion with soda and borax, gives a tolerably malleable metallic grain, which is very magnetic.

After roasting, it behaves with the fluxes like oxide of nickel, and generally gives a glass, which is slightly blue, owing to the presence of a small quantity of cobalt.

ON THE DISCRIMINATION OF MINERALS BY MEANS OF THE BLOW-PIPE, AIDED BY HUMID ANALYSIS.

We now come to that part of our subject which treats of the discrimination of minerals by a few simple tests;

and for the greater part of the remarks which follow, the author is indebted to Campbell's translation of Kobell's work on this subject, and to Chapman's Practical Mineralogy; and for a more extended account of these processes, the reader is referred to the above-mentioned works.

In both the works just quoted, the first thing to be done with a mineral in its examination, is to ascertain whether its lustre be *metallic* or *non-metallic*; this point is most important. There are, however, a few minerals in which the determination of this is doubtful; in which case, a single experiment will point out to which class the mineral really belongs.

The *degree of fusibility* is the next thing to be attended to; and in case a beginner should be mistaken on this point, Von Kobell has arranged a certain set of minerals to be employed as checks against the opinion any one may form of the fusibility of any substance submitted to his examination.

They are six in number, and are as follows:

1. Sulphuret of antimony.
2. Natrolite.
3. Almandine.
4. Strahlstein (actinolite).
5. Adularia (felspar).
6. Diallage (bronzite).

The great advantage of this scale is, that it facilitates the very useful practice of comparative experiment. If a mineral be more fusible than No. 2, and less so than No. 3, it is said to be 2.5 in the scale of fusibility, and so on.

Another great requisite, is to ascertain the *hardness* of any specimen we may have to examine; and here again a comparative scale is necessary. The following has been arranged by Mohs:

1. Talc.
2. Gypsum (crystallized).
3. Calc spar (carbonate of lime).
4. Fluor spar.
5. Apatite.
6. Adularia (felspar).
7. Rock crystal.
8. Topaz.
9. Corundum.
10. Diamond.

As the above set of minerals may not always be at hand, and as it is very necessary to determine the degree of hardness, Mr. Chapman has, with much ingenuity, contrived a very useful set of materials which are always present. This scale is of great value to the experimenter.

1. Yields easily to the nail.

2. Yields with difficulty to the nail, or merely receives an impression from it. Does not scratch a copper coin.

3. Scratches a copper coin; but is also scratched by it, being of about the same degree of hardness.

4. Not scratched by a copper coin; does not scratch glass.*

* Mr. Chapman has here omitted an important observation; he has not stated what particular kind of glass, as that substance varies so much in hardness.

5. Scratches glass, though rather with difficulty, leaving its powder on it. Yields readily to the knife.

6. Scratches glass easily. Yields with difficulty to the knife.

7. Does not yield to the knife. Yields to the edge of a file, though with difficulty.

8. 9. 10. Harder than flint.

In ascertaining the degrees of hardness by the first scale, a file ought to be employed, and it is by the comparative ease or difficulty that the mineral and sample mineral yield to it that the degree of hardness is determined. This is a part, however, to which much attention must be paid.

The *specific gravity* of a mineral is also of great importance in its discrimination. It may be determined as follows: if the sample be of sufficient size to suspend from the pan of a balance by means of a fibre of silk; if not, another mode must be adopted, which will be pointed out as we proceed.

If the mineral can be suspended, attach it by a short fibre of silk to one of the pans of a delicate balance, and ascertain its weight; then immerse it (still suspended to the pan) in distilled water of the temperature of 60° Fahr., and then note its weight; it will be found to have lost a certain amount, which will correspond to the weight of the bulk of water it has displaced. Divide its weight in air by the loss of weight in water, and the quotient will be the required specific gravity. This will be more readily understood by an example. Suppose we find the mineral to weigh 80 grains in the air, and only 66 in water; the loss $= 80 - 66 = 14$. We must now divide 80 by 14, thus $\frac{80}{14} = 5.7$, which

is about the specific gravity of a sample of bournonite.

We have now the second case to consider; the mineral may be in very small fragments, or it may even be in powder, in which case its specific gravity must be determined thus:

A bottle must be obtained capable of holding a known quantity of water, as 500 grs. The ordinary specific gravity bottle is well suited for this purpose. A known weight of the body whose specific gravity is to be ascertained, say 100 grs. is to be introduced into the bottle, which is then to be filled with water, and the weight of the whole to be determined, it will be less than the united weight of the contents of the bottle full of water and the weight of the mineral in air. The loss of weight is to be ascertained, and the weight in air divided by it; the quotient is the specific gravity required. The following example may suffice to render this clearer:

Suppose the bottle contains of water .	500 grs.
And the mineral introduced into it weighs	100 grs.
	600
Weight of mineral and water in bottle .	560
	40 loss.

Then $\frac{100}{40} = 2.5$ required specific gravity.

Besides ascertaining hardness, fusibility, and specific gravity, it is required to determine the re-actions of the mineral with the various fluxes. The method of doing this has already been described. Finally, it is necessary to ascertain the behaviour of the mineral

in solution, or otherwise, with certain liquid re-agents. The method of conducting this class of examination will be pointed out in a following chapter.

Having thus given a rough sketch of the processes to be employed, we will now describe the whole method to be followed in ascertaining the nature of certain minerals, principally confining ourselves to those generally met with, and worked by the miner. The following is a list of them.

Amalgam.
Anhydrite.
Anhydrous silicate of zinc.
Grey antimony.
Native antimony.
Native mercury.
Red antimony.
Oxide of antimony.
Antimonial nickel.
Antimonial silver.
Antimonial grey copper.
Dark red silver.
Muriate silver, (horn silver.)
Native silver.
Sulphuret of silver.
Argentiferous gold.
Argentiferous grey copper.
Arseniate of cobalt.
Arseniate of copper.
Arseniate of iron.
Arseniate of lead.
Native arsenic.
Arsenic glanæ.
Oxide of arsenic.
Red sulphuret of arsenic.
Yellow ditto.
Arsenical iron.
Arsenical cobalt.
Arsenical nickel.
Arsenical pyrites.
Arsenical grey copper.
Arsenical bismuth.
Arsenious acid.
Arsenic acid.
Light-red silver.
Muriate of copper, (Atakamite.)
Azure copper ore.
Azurite.
Azure stone.
Carbonate of baryta.
Sulphate of baryta.
Bismuth blende.
Native bismuth.

Black manganese.
Black oxide of copper.
Black silicate of manganese.
Black tellurium.
Graphic tellurium.
Galena.
Sulphate of lead.
Carbonate of lead.
Horn lead, (chloride of lead.)
Blende.
Bog iron ore.
Bournonite.
Bright-white cobalt.
Calamine.
Calomel.
Baryta, carbonate.
Copper, carbonate, green.
Copper, carbonate, blue.
Iron, carbonate.
Lime, carbonate.
Silver, carbonate.
Strontian, carbonate.
Phosphate of lime.
Chromate of iron.
Chromate of lead.
Cinnabar.
Copper.
Copper pyrites.
Copper, red oxide.
Copper, vitreous.
Earthy cobalt.
Oxide of tin.
Sulphuret of tin.
Oligistic iron.
Iron, hydrated oxide.
Iron, spathose.
Iron, specular.
Iron, sulphuret.
Iron, sulphuret, white.
Iron, sulphuret, magnetic.
Fluor spar.
Galena.
Graphic tellurium.
Gypsum.
Gypsum, anhydrous.
Hydrated deutoxide of manganese.
Hydrous silicate of iron.
Nickel, arsenical.
Nickel ochre.
Tin stone.
Palladium.
Platinum.
Purple copper.
Quartz.
Sulphuret of molybdenum.
Sulphuret of nickel.
Sulphuret of nickel and bismuth.

CLASS I. MINERALS POSSESSING A METALLIC LUSTRE.

CLASS II. METALS POSSESSING NO METALLIC LUSTRE.

These classes are further divided thus :

CLASS I.

Division 1. *Metals proper.*

Division 2. *Fusible* 1-5, *or readily volatile.*

Division 3. *Infusible, or fusibility above* 5, *and not volatile.*

CLASS II.

Division 1. *Easily volatile or combustible.*

Division 2. *Fusible* 1-5 ; *not, or only partially volatile.*

Division 3. *Infusible, or fusible above* 5.

Division 1. CLASS I.

Metals proper, readily distinguished.

Division 2. CLASS I.

Section 1. Give an arsenical odour on charcoal.

Section 2. Give the horse-radish odour of selenium.

Section 3. Give a white sublimate in open glass tube, which is fusible into colourless drops, indicative of tellurium.

Section 4. On charcoal give antimonial vapour.

Section 5. With soda give a sulphuret, but do not give indications as above.

Section 6. Behave differently to five preceding.

Division 3. CLASS I.

Section 1. Give the re-action of manganese with borax.

Section 2. On charcoal in reducing flame become magnetic.

Section 3. Partly agreeing with section 2.

CLASS II.

Division 1. Easily volatile or combustible.

Division 2. *Part* 1. *Give on charcoal, either alone or with soda, a metallic bead or magnetic metallic mass.*

Section 1. Give silver.

Section 2. Give lead.

Section 3. Give copper, and when moistened with hydrochloric acid, colour the flame transiently blue.

1. Give a strong odour of arsenic.

2. Give no odour of arsenic.

Section 4. Give a bright blue with borax.

Section 5. Give a black or grey metallic magnetic mass, but not behaving as any of the minerals of the preceding sections.

1. Indicate arsenic during fusion.

Section 6. Not belonging to either of the foregone sections.

Part. 2. *Give no metallic bead, or magnetic metallic mass.*

Section 7. After fusion, alone or with charcoal, have an alkaline reaction.

Division 3. CLASS II.

Section 1. Ignited with nitrate of cobalt give zinc re-action.

Section 2. Soluble in hydrochloric or nitric acids, giving no jelly or residue.

Section 3. Not included in the five preceding sections, but divided thus:

1. Hardness under 7.

2. Hardness 7, or above 7.

CLASS. I.

Division 1. *Metals proper.*

Native mercury.—Liquid at ordinary temperatures. Tin-white and opaque. Lustre metallic. Sp. gr. 12. Entirely volatilizable before the blow-pipe.

Native silver.—Hardness = 2.5 — 3. Silver-white, perfectly malleable, easily fusible, soluble in nitric acid. The solution, mixed with hydrochloric acid, gives a white curdy precipitate of chloride of silver, which is soluble in ammonia. Metallic silver is also precipitated from the nitric acid solution by a plate of copper. Sp. gr. about 10.

Native Gold.—Hardness, 2.5 — 3. Colour, yellowish or greyish yellow, easily fusible and very ductile, soluble in nitro-hydrochloric acid, (aqua regia), without residue, the solution giving a purple precipitate on the addition of protochloride of tin, and a brown precipitate with protosulphate of iron. Sp. gr. about 19.

Electrum, Auriferous Silver.—Colour, inclining to silver-white. Hardness, 2.5 to 3. Sp. gr. about 12.5. Partially soluble in aqua regia, giving a residue of chloride of silver. The solution is acted on by protosulphate of iron, as the above.

Native Iron.—Hardness, 4.5. Light steel-grey, ductile and malleable, attractible by the magnet, soluble in dilute sulphuric acid, giving a blue precipitate with ferrocyanide of potassium. Sp. gr. 7.4 to 7.8.

Native Copper.—Colour brownish red, ductile, and malleable. Hardness, 2.5 to 3. Sp. gr. 8.4 to 8.9, easily soluble in nitric acid, with evolution of red fumes. The solution is blue, which becomes intense on the

addition of excess of ammonia. Metallic copper can be obtained from it by precipitation on a plate of iron or zinc.

Native Lead.—Hardness, 1.5. Sp. gr. 11 to 12. Colour, lead-grey, ductile and malleable, easily fusible, giving a yellow sublimate of oxide of lead on the charcoal support. Soluble in nitric acid, the solution giving a white precipitate with sulphuric acid, or yellow with chromate of potash, and a deeper yellow with iodide of potassium. Metallic lead is also obtainable from it by deposition on a bar of zinc.

Native Platinum.—Hardness, 4 to 4.5. Sp. gr. 16 to 20. Colour, silver-whitish or grey, ductile and malleable. Not fusible, or acted on by any fluxes. Soluble in aqua regia. The solution gives a yellow granular precipitate with carbonate of potash, which is insoluble in excess.

Native Palladium.—Hardness 4.5 to 5. Sp. gr. 11.5 to 12.5. Colour, steel-grey or silvery white. Soluble in nitric acid. The solution gives a brownish precipitate with carbonate of potash, which is soluble in excess. The metal also becomes blue when heated gently in the oxidating flame, but which colour disappears when the heat is more intense. It is infusible by itself, but fusible with sulphur, which on being burnt off, leaves a globule of palladium.

Division 2. CLASS I.

Section 1. *Give a strong odour of arsenic.*

Native Arsenic.—Hardness, 3.5. Sp. gr. 5.7 to 5.9. Colour, tin-white on fresh fracture, but soon tarnishes. Fusible, entirely volatilizable if pure.

Arsenical Grey Copper.—Hardness, 3 to 4. Sp. gr. 4.4 to 5. Moistened with hydrochloric acid and ignited on charcoal, gives an intense blue colour to the flame. It gives a grey globule, generally attractible by the magnet, and a bead of copper with borax. Acted on by caustic potash, the sulphurets of antimony and arsenic are dissolved out, and may be precipitated from the solution by hydrochloric acid. Some varieties contain silver, the presence of which may be ascertained by acting on the ore with nitric acid, and precipitating with common salt, or hydrochloric acid. The precipitate ought to be soluble in ammonia.

Tin-white cobalt.—Hardness 5.5. Sp. gr. 5.4 to 6.7. Colour, tin-white; gives blue colour to borax, and pink solution to nitric acid. Silicate of potash in solution gives a sky-blue precipitate. It fuses into brittle magnetic globules.

Bright-white Cobalt.—Hardness 5.5. Sp. gr. 6.2 to 6.3. Colour, silver-white, sometimes reddish. Roasted in the open tube it gives off sulphurous acid, which may be ascertained by smell and test paper. It fuses into a magnetic globule, giving cobalt blue to borax. Its solution in nitric acid behaves with silicate of potash as before, but gives a copious precipitate with nitrate of baryta, indicative of the presence of sulphuric acid.

Arsenical Nickel, Nickeline.—Hardness, 5 to 5.5. Sp. gr. 6.6 to 7.7. Colour, reddish or whitish. Fuses readily into a white or yellowish globule. Partially soluble in nitric acid. The solution is green, and with silicate of potash, gives an apple-green precipitate. The solution becomes violet-blue on the addition of an

excess of ammonia. In the varieties of this mineral containing sulphur, the nitric acid solution gives a white precipitate, with nitrate of baryta, or chloride of barium.

Arsenical Iron.—Sp. gr. 6.2. Hardness, 5 to 5.5. Colour, steel-grey. Fuses into a brittle magnetic globule, which gives the re-actions of iron with the fluxes. It is soluble in nitric acid, the solution giving a blue colour to ferrocyanide of potassium, and a yellowish brown precipitate of arseniate of iron to ammonia.

Section 2. *Give evidence of the presence of selenium.*

Section 3. *Give evidence of the presence of tellurium.*

Graphic tellurium.—Hardness, 1.5 to 2. Sp. gr. 5.7. Steel-grey, like galena. Easily fusible, giving a yellowish white button, which, on lamination, is partially soluble in nitric acid; the solution containing silver, which can be ascertained by chloride of sodium. The residue is soluble in aqua regia, and gives all the characteristics of a solution of gold. It gives no precipitate with sulphuric acid.

Black Tellurium.—Hardness, 1 to 1.5. Sp. gr. 7 to 7.2. Blackish lead-grey. Very fusible, producing a bead of malleable metal. The charcoal gives evidence of the presence of lead by the coating of yellow oxide. It dissolves in nitric acid, and the solution gives a dense white precipitate with sulphuric acid.

Section 4. *Give evidence of antimony.*

Native Antimony.—Sp. gr. 6.5 to 6.8. Hardness, 3 to 3.5. Colour, tin-white, but becomes yellowish by exposure before the blow-pipe. It fuses readily, and continues to burn for some time after it has been removed from the flame; the globule being covered with crystals.

Grey Antimony, Sulphuret of Antimony.—Sp. gr. 4.3 to 4.6. Hardness, 2. Colour, light grey. It melts even in the flame of a candle. It is absorbed by charcoal before the blow-pipe. If heated with solution of caustic potash, its powder becomes ochry red, and almost entirely dissolves. It can be re-precipitated by hydrochloric acid.

Bournonite.—Sp. gr. 5.7 to 5.8. Hardness, 2.5 to 3. Colour approaching to steel-grey, with a shining lustre. Before the blow-pipe it decrepitates. A crust of sulphuret of lead remains after fusion, enclosing a globule of copper. It partially dissolves in nitric acid. The solution is blue, and gives with sulphuric acid a white precipitate of sulphate of lead, and an azure blue with ammonia.

Antimonial Silver.—Sp. gr. 9.4 to 9.8. Hardness, 3.5. Colour, between silver-white and tin-white. Before the blow-pipe, on charcoal, it melts into a grey metallic globule, which is not malleable. It gives no sulphuret with soda, and is not acted on by caustic potash; with borax and soda it gives a button of silver.

Antimonial Grey Copper.—Its colour is dark red grey. Sulphuret of antimony can be obtained from it by the action of caustic potash. It gives evidence of sulphur with soda, and its solution in nitric acid gives a deep blue with ammonia. It gives with borax and soda a button of silver.

Antimonial Nickel.—Sp. gr. 6.4 to 6.5. Hardness, 5 to 5.5. Colour, steel-grey or silver-white. Before the blow-pipe, is partly volatilized, and finally gives a metallic globule, which is magnetic. It is readily dissolved in nitric acid, giving the usual indication of nickel. It is scarcely acted upon by hydrochloric acid.

Section 5. *Give a sulphuret with soda, but do not possess the general properties of the foregone sections.*

Sulphuret of Silver.—Sp. gr. 6.9 to 7.2. Hardness, 2 to 2.5. Dark lead-grey colour. In the flame of a candle it intermesces. It is sectile. Before the blow-pipe the sulphur volatilizes, and a bead of pure silver remains. It is soluble in dilute nitric acid, and gives the characteristic curdy precipitate (which is soluble in ammonia) by the re-action of hydrochloric acid.

Sulphuret of Manganese.—Sp. gr. 3.9 to 4. Hardness, 3.5 to 4. Colour, brownish black, streak dark green; this is almost characteristic. It gives the usual indications of manganese by the blow-pipe with the fluxes. It dissolves, when in powder, in hydrochloric acid with the evolution of sulphuretted hydrogen.

Vitreous Copper. — Sp. gr. 5.6 to 5.8. Hardness, 2.5 to 3. Colour, lead or iron-grey. Before the blow-pipe on charcoal, it gives off sulphurous acid, and when the sulphur is wholly volatilized gives a bead of copper. With nitric acid it gives a green solution, which becomes azure-blue on addition of ammonia. Moistened with hydrochloric acid, the flame is coloured blue.

Sulphuret of Tin, (Tin Pyrites).—Sp. gr. 4.3 to 4.7. Hardness, 4. Steel-grey colour. Before the blow-pipe sulphur is driven off; it then fuses to a black slag. Soluble in nitric acid, with separation of sulphur and oxide of tin. The solution gives a deep blue with ammonia. This mineral gives, by itself, no bead of malleable metal.

Sulphuret of Copper, (*Copper Pyrites.*)—Sp. gr. 4.1 to 4.3. Hardness 3.5 to 4. Colour, brass-yellow. It fuses on charcoal before the blow-pipe, and melts into a black brittle globule, attractible by the magnet. With borax, in small proportion, it yields a copper bead, and when the blast is long continued, gives *per se* a button of copper or charcoal. It is soluble in nitric acid under separation of sulphur.

Purple Copper.—Sp. gr. 5. Hardness, 3. Colour, between copper-red and brown. Soft, and easily frangible. Before the blow-pipe it blackens and becomes red on cooling; at a higher temperature it fuses, the globule is magnetic. Fused with soda, it is reduced with the formation of a bead of copper. It is soluble in nitric acid. The solution acted on by ammonia gives a precipitate of peroxide of iron and an azure-blue solution. The original solution gives a white precipitate with chloride of barium.

Sulphuret of Nickel.—Sp. gr. 6.4. Hardness, 4. It is steel-grey. It gives, with a strong continued heat, a globule which is magnetic, malleable and metallic. In the open tube it gives sulphurous acid. It is not much acted on by nitric acid, but dissolves in aqua regia, giving a greenish solution, in which potash and silicate of potash give an apple-green precipitate, and chloride of barium a white precipitate.

Sulphuret of Iron.—Sp. gr. 4.7 to 5. Hardness 6 to 6.5. General colour, brass-yellow; does not yield to the knife; hence it may be distinguished from copper pyrites. It does not attract the magnet before roasting, but does after; it is only acted on very slightly by muriatic acid, but is decomposable by aqua-regia. The

solution gives indications of sulphur and iron, by chloride of barium and ferrocyanide of potassium.

Magnetic Iron Pyrites.—Sp. gr. 4.4 to 4.7. Hardness, 3.5 to 4.5. General colour, bronze-yellow. Acts on the magnet before being heated. Dissolves very readily in hydrochloric and dilute sulphuric acids, under evolution of sulphuretted hydrogen.

Sulphuret of Bismuth.—Sp. gr. 6.5. Hardness, 2 to 2.5. Colour, tin-white, or lead-grey. It melts in the flame of a candle, before the blow-pipe; the greater part of it is volatilized. It gives a bead of bismuth, and coats the charcoal with the oxide of that metal. It is soluble in nitric acid, with a separation of sulphur; the solution lets fall a white precipitate on dilution with water.

Sulphuret of Lead (galena).—Sp. gr. 7.4 to 7.6. Hardness, 2.7. Colour, lead-grey. Before the blow-pipe, it decrepitates; after long heating, it is reduced to metallic lead, which on cupellation generally yields a little silver. It is soluble in dilute nitric acid; the solution gives metallic lead on a slip of zinc, a white precipitate with sulphate of soda, and a yellow one with iodide of potassium.

Section 6. *The following minerals belong to this division, but cannot be readily classed.*

Amalgam.—Sp. gr. 10 to 14. Hardness, 1 to 3.5. Colour, silver-white, or greyish. It is sometimes fluid, sometimes solid. Before the blow-pipe it volatilizes, and a globule of silver remains. It is easily soluble in nitric acid, and whitens the surface of copper when rubbed upon it; in the mattrass, sublimed mercury is obtainable.

Native Bismuth.—Sp. gr. 9.6 to 9.8. Hardness, 2 to 2.5. Colour, silver-white, tinged with red. When cold, it is not malleable; but when hot, may be laminated. It readily fuses and vaporizes, coating the charcoal with an orange-yellow sublimate. It dissolves readily in nitric acid.

Black Silicate of Manganese fuses with intermescence, and gives a deep amethyst-red with borax; it gives a large quantity of water in the mattrass. Its general colour is lead-grey to iron-black. It gives a green colour to soda on the platinum foil. It is soluble in muriatic acid, with disengagement of silica.

Division 3. CLASS I.

Section 1. *Give the reactions of manganese with borax.*

All the oxides of manganese belong to this class; they may be distinguished by their action, as above stated, with borax, in conjunction with the fact that they evolve chlorine when treated with hydrochloric acid, and the solution gives a flesh-red precipitate with hydrosulphuret of ammonia, and a dirty-white with caustic potash, which speedily becomes brown. They also give the characteristic green when acted on by soda on the platinum foil. There are several varieties; but it does not come within our province to particularize them.

Section 2. *On charcoal, in reducing flame, becoming magnetic.*

Red Hæmatite.—There are several varieties of this ore; some crystalline, others not; those which are not, have a very low degree of hardness, which is not determinate; those which are, have a hardness of from 5.5

to 6. They may be distinguished by becoming magnetic after roasting, and giving the usual indication of iron with the fluxes. They are slightly soluble in hydrochloric acid; but if fused with carbonate of soda, they are perfectly soluble in that acid. The solution, after boiling with a few drops of nitric acid, gives a deep blue with ferrocyanide of potassium, and a brown bulky precipitate, with an excess of ammonia or caustic potash.

Section 3. *Partially agreeing with the above minerals.*

Magnetic Iron Ore.—This ore, before roasting, is strongly attracted by the magnet, as its name implies. It slowly dissolves in strong hydrochloric acid. Its colour is iron-black, with a shining metallic or glimmering lustre. It colours glass of borax, in the oxidating flame, deep red, which becomes dingy yellow on cooling. In the reducing flame, it is bottle-green.

Chrome Iron Ore.—Sp. gr. 4.3 to 4.6 Hardness, 5.4. It is iron-black, or brownish black, with a shining and somewhat metallic lustre. Some varieties are strongly magnetic. With borax and microcosmic salt, it melts slowly, and the beads possess the fine green colour imparted by chromium, which increases in intensity on the addition of tin. It is scarcely acted on by acids. It gives a yellow solution to water after fusion with nitre; the solution gives a green with alcohol and sulphuric acid, or with sulphuretted hydrogen.

Sulphuret of Molybdenum.—Sp. gr. 4.4 to 4.7. Hardness, 1 to 1.5. Its colour is similar to that of freshly cut metallic lead. It is opaque, highly sectile and flexible. It is unctuous to the touch. Held in the forceps, it colours the blow-pipe flame light green. It

is acted on by nitric acid, with effervescence, and leaves a grey substance, which is molybdic acid; if this be treated with alcohol and sulphuric acid, heated and allowed to cool, it becomes a splendid intense blue, which entirely disappears on the addition of water.

CLASS II.

Minerals possessing no metallic lustre.

Division 1. *Easily volatile or combustible.*

Sulphur.—Sp. gr. about 2. Hardness, 1.5 to 2.5. Its colour, when pure, is the well known sulphur yellow, that is, in a state of powder, but when in the crystalline form, is more of a deep topaz yellow. It is, however, from extraneous circumstances and mixtures, sometimes grey, brown, or even greenish. It burns, giving off the well-known smell of burning sulphur.

Realgar, Red Sulphuret of Arsenic.—Sp. gr. 3.3 to 3.6. Hardness, 1.5 to 2. It is of a bright red colour, approximating to scarlet, sometimes having an orange tint. Before the blow-pipe, on charcoal, it burns with a pale yellow flame, giving an odour of garlic. It is soluble in caustic potash, from which hydrochloric acid precipitates lemon-yellow flocks.

Orpiment, Yellow Sulphuret of Arsenic.—Sp. gr. 3.45. Hardness, 1.5 to 2. It behaves like realgar, but is distinguished by its fine yellow colour.

Arsenious Acid.—Sp. gr. 3.6 to 3.7. Hardness, 1.5. Colour, snow-white, though occasionally tinged with red or brown: this is accidental. It is soluble in hot water; the solution giving a yellow precipitate with sulphuretted hydrogen. It sublimes on the mattrass;

the deposit is crystalline. On charcoal, it exhales the odour of garlic in the reducing flame.

Oxide of Antimony.—Sp. gr. 5.5. to 5.6. Hardness, 2.5 to 3. Its colour is generally snow-white, although sometimes yellow or grey. It melts very readily before the blow-pipe, and is volatilized as a white vapour, very characteristic of antimony. It is entirely soluble in hydrochloric acid without the evolution of any gas, and does not change its colour in caustic potash. With borax it forms a bead, which is yellowish while hot, but colourless when cold.

Red Antimony.—Sp. gr. 4.5 to 4.6. Hardness, 1 to 1.5. It is cherry-red, fuses easily on charcoal, which absorbs it; it is, however, finally volatilized. If immersed in nitric acid, it becomes covered with a white coating. It is soluble in hydrochloric acid, with the evolution of sulphuretted hydrogen. Its colour is changed to ochre-yellow by immersion in caustic potash.

Sulphuret of Mercury, Cinnabar.—Sp. gr. 6.7 to 8.2. Hardness, 2 to 2.5. Its colour varies from carmine to vermilion red, or lead-grey. It always gives a bright scarlet streak. It fuses before the blow-pipe, and volatilizes with a blueish flame and sulphurous odour. Mixed with soda, in the mattrass, it gives metallic mercury. Its colour is not changed by the ordinary acids or by caustic potash.

Chloride of Mercury, Horn Mercury, Calomel.—Sp. gr. 6.4 to 6.5. Hardness, 1.5 to 2. Colour, greyish-white, grey, or yellowish. It is completely volatilized before the blow-pipe, and gives metallic mercury when

heated in the mattrass with soda. If treated with caustic potash, it becomes black instantaneously.

Division 2. *All minerals having no metallic lustre and exhaling the odour of arsenic before the blow-pipe, belong to this division, with the exception of arseniate of lime.*

Part 1. *Give on charcoal, either alone or with soda, a metallic bead or magnetic metallic mass.*

Section 1. *Give Silver.*

Light Red Silver.—Sp. gr. 5.4 to 5.6. On charcoal gives the garlic odour, characteristic of arsenic. If the pulverized mineral be treated with caustic potash, the solution gives lemon-yellow flocks of sulphuret of arsenic, on the addition of hydrochloric acid.

Dark Red Silver.—Sp. gr. 5.8 to 5.9. On charcoal it gives the antimonial fume, and with caustic potash, its solution gives orange-red flocks of sulphuret of antimony, on the addition of hydrochloric acid.

Horn Silver.—Sp. gr. 4.7 to 5.5. Colour, pearl-grey, greenish or reddish blue. It is fusible in the flame of a candle. When rubbed with a piece of moistened zinc, its surface becomes covered with a thin film of silver. It is insoluble in nitric acid. If treated before the blow-pipe, with a bead of microcosmic salt, in which oxide of copper has been dissolved, it colours the flame a splendid purple.

Section 2. *Give Lead.*

Arseniate of Lead.—Sp. gr. 6.9. to 7.3. Hardness, 3.5 to 4. Its colour varies from many shades of yellow to aurora or hyacinth red. Before the blow-pipe it fuses with difficulty, giving off arsenical fumes. It is

soluble in nitric acid; its solution gives metallic lead to a plate of zinc, and a white precipitate with sulphuric acid or a soluble sulphate. When pure, it dissolves without residue in caustic potash.

Sulphate of Lead.—Sp. gr. 6.2 to 6.3. Hardness, 3. General colour, white grey, or yellowish.

It decrepitates before the blow-pipe; fuses in the oxidating flame to a transparent bead, which becomes white on cooling. It gives a sulphuret with soda. It dissolves in caustic potash, and with difficulty in nitric acid.

Carbonate of Lead.—Sp. gr. 6.3 to 6.6. Hardness, 3 to 3.5. Either colourless or white, passing into a greyish black. Its powder, thrown on coal, emits a phosphorescent light. Before the blow-pipe it decrepitates, becomes yellow, and then red, and finally becomes a bead of metallic lead. It is soluble, with effervescence, in nitric acid, and is also soluble in caustic potash. Its acid solution gives a fine yellow with chromate of potash or iodide of potassium.

Chloride of Lead, Horn Lead.—Sp. gr. 7 to 7.1. Hardness, 2.5 to 3. Before the blow-pipe it is reduced, giving off hydrochloric acid. With microcosmic salt and oxide of copper, it gives a beautiful blue flame before the blow-pipe. It dissolves in nitric acid and caustic potash.

Chromate of Lead.—Sp. gr. 5.9 to 6.0. Hardness, 2.5. Colour, deep red, or hyacinth red. Its lustre is adamantine and streak yellow; before the blow-pipe it becomes black and decrepitates. It gives an emerald green colour to borax, and dissolves in hydrochloric acid, with separation of chloride of lead. It is soluble

without effervescence, in nitric acid, giving a yellowish solution. When fused with nitre, it gives a yellow solution in water, which is chromate of potash.

Section 3. *Give copper, and when moistened with hydrochloric acid, colour the flame transiently blue.*

1. *Give a strong odour of arsenic.*

2. *Give no odour of arsenic.*

Arseniate of Copper.—There are several varieties of arseniate of copper belonging to this section, which need not be particularized. Arseniate of copper may be detected by boiling it with caustic potash; there is a separation of oxide of copper, and the arsenic acid unites with the potash, forming arseniate of potash, which gives, with sulphuretted hydrogen a yellow precipitate, and with nitrate of silver a brick-red precipitate.

2. *Give no odour of arsenic.*

Chloride of Copper.—Sp. gr. 4 to 4.3. Hardness, 3 to 3.5. Colours, various shades of green. It tinges the flame of the blow-pipe or a candle bright blue. It is soluble, without effervescence, in nitric acid, and gives to ammonia a bright azure blue.

Blue Carbonate of Copper.—Sp. gr. 3.5 to 3.7. Hardness, 3. to 4. Colour, from azure to Berlin blue, with an occasional tinge of black. It yields easily to the knife. Before the blow-pipe, it decrepitates, blackens, and ultimately fuses. It is soluble, with effervescence, in nitric acid, and the solution, on the addition of ammonia in excess, becomes azure blue. All solutions of copper deposit that metal on a plate of clean iron.

Green Carbonate of Copper, Malachite.—Sp. gr. 3.5 to 4. Hardness, 3.5 to 4. Colour, various shades of

green. Before the blow-pipe it decrepitates, and fuses into a black slag, and behaves as the blue carbonate.

Black Oxide of Copper dissolves readily in acids. This mineral rarely occurs massive.

Red Oxide of Copper, Ruby Copper.—Sp. gr. 5.6 to 6.1. Hardness, 3.5 to 4. The colour of this mineral is red, of various shades. Before the blow-pipe, it is reducible, like the last species, on charcoal, to the metallic state. It acts as under with the fluxes. Soluble in nitric acid. Its solution in hydrochloric acid gives a white precipitate with water, and an ochre-yellow precipitate with caustic potash. Under similar circumstances, the solution of black oxide gives no precipitate with water, and a blueish precipitate with caustic potash.

Section 4. *Give a bright blue with borax.*

Arseniate of Cobalt.—Sp. gr. 2.9 to 3. Hardness, 2.5 to 3. Colour, reddish violet, or red; gives water by calcination. Fusible in the blow-pipe flame, with disengagement of arsenical vapour; soluble in nitric cid. The rose-coloured solution gives with the alkalies a violet-coloured precipitate; and with ferrocyanide of potassium, a green precipitate.

Nickel Ochre.—This substance is found adhering to or coating arsenical nickel, and is doubtless derived from its decomposition.

General colour, apple green. In the mattrass, it loses about one-fourth of its weight of water. It is soluble in acids, without effervescence. Caustic ammonia in excess gives a blue solution. It gives a strong arsenical odour before the blow-pipe on charcoal.

Section 5. *Give a black or grey metallic mass, but not*

behaving as any of the minerals of the preceding sections.

1. *Indicate arsenic during fusion.*

Arseniate of Iron.—Sp. gr. 2.9 to 3. Hardness, 2.5. Various shades between light and dark green, or yellowish and brownish green. Before the blow-pipe, on charcoal, it gives off arsenical vapours, and fuses into a grey slag, which exhibits the metallic lustre, and is attracted by the magnet. It gives, after roasting, bottle-green globules with the fluxes.

Section 6. *Not belonging to either of the foregone sections.*

Bismuth Blende.—Sp. gr. 5.9 to 6. Hardness, 3.5 to 4. Colour, dark brown to wax yellow. Decrepitates before the blow-pipe, giving off an arsenical odour, and is ultimately converted into a glass, which effervesces with borax. It is reduced with soda, on charcoal, to a button of bismuth. It forms a perfect jelly with hydrochloric acid, and its solution gives a white precipitate on the admixture of water.

Part 2. *Give no metallic bead or magnetic metallic mass.*

Section 7. *After fusion, alone, or with charcoal, have an alkaline re-action.*

Anhydrite.—Sp. gr. 2.5 to 2.9. Hardness, 3 to 3.5. The colour varies from white to blueish violet or reddish; it is translucent, and sometimes transparent. In the mattrass it gives no water. Before the blow-pipe, it becomes glazed over with a whitish enamel. Anhydrite is sparingly soluble in water; the solution gives with oxalate of ammonia a white precipitate, and the same

with a soluble salt of baryta. It dissolves quietly in a tolerably large quantity of hydrochloric acid.

Gypsum behaves as above, with the exception that it gives water; and instead of becoming glazed with a white enamel before the blow-pipe it exfoliates.

Carbonate of Baryta.—Sp. gr. 4.3. Hardness, 3 to 3.5. Exposed to the blow-pipe flame, in the platinum forceps, it melts readily into a white enamel, with a brilliant light. It dissolves in diluted hydrochloric or nitric acid; the solution gives a precipitate with solution of sulphate of lime, or any other soluble sulphate.

Sulphate of Baryta.—Sp. gr. 4.4 to 4.6. Hardness, 3 to 3.5. Its colour varies much, being shades of grey, red, green, or yellow; its general colour is, however, white; it is either transparent or opaque. When fused in the platinum tongs, it imparts a greenish yellow colour to the blow-pipe flame. It decrepitates, and is difficultly fusible, but finally melts into a white enamel. If fused with carbonate of soda, the watery solution gives a precipitate with a salt of baryta, as do all the earthy insoluble sulphates.

Sulphate of Strontia, Celestine.—Sp. gr. 3.6 to 4. Hardness, 3 to 3.5. It is white, grey, yellow, or reddish; it also occurs of a beautiful delicate blue, hence the term celestine has been applied to it. It decrepitates before the blow-pipe, and behaves like the preceding mineral, sulphate of baryta, with this exception, that it colours the blow-pipe flame, faintly, reddish purple. This test, however, can be rendered more delicate by moistening the specimen with hydrochloric acid, before submitting it to the blow-pipe; the colour of the flame is then intensely purple.

Fluor Spar, Fluoride of Calcium.—Sp. gr. 3 to 3.3. Hardness, 4. This mineral appears of many colours, as white, yellow, purple, and green. If it be pulverized and placed on a red hot plate of iron or glowing coals, it phosphoresces. It is acted on very powerfully by hot sulphuric acid, and evolves vapours of hydrofluoric acid, which corrode glass. The best method of ascertaining the presence of this mineral, is to heat it in a glass tube with bisulphate of potash; the vapours given off corrode glass, as above stated, and turn Brazil-paper yellow.

Division 3. CLASS II.

Section 1. *Ignited with nitrate of cobalt, give zinc re-action.*

Calamine.—Sp. gr. 4.2 to 4.5. Hardness, 5. Its general colour is greyish or yellowish; but it sometimes occurs of various shades of green or brown. It dissolves with effervescence in nitric or hydrochloric acid. It is infusible before the blow-pipe; but after ignition, it behaves like oxide of zinc. On charcoal, it is reduced, coating the charcoal with a white sublimate, which when hot is yellow.

Section 2.

Carbonate of Iron, Spathose Iron.—Sp. gr. 3.6 to 3.8. Hardness, 3.5 to 4.5. Colour, various shades of yellow; when exposed to the action of the atmosphere, it becomes brownish. It dissolves in hydrochloric acid, with effervescence, arising from the escape of carbonic acid. The solution gives a blue with ferrocyanide of potassium, and a whitish green with caustic potash, which speedily becomes brown, more especially on the

surface. It affects the magnetic needle. It communicates to borax a bottle-green colour. The *hydrous oxide of iron, bog iron ore, clay iron stone,* &c., behave in much the same way, excepting that the two first do not effervesce with hydrochloric acid.

Earthy Cobalt.—Sp. gr. 2.1 to 2.4. Very soft. Its colour varies from brown to black. It exhales a slight arsenical odour before the blow-pipe, and forms a deep-coloured blue glass with borax.

Zinc Blende, Sulphuret of Zinc.—Sp. gr. 4 to 4.2. Hardness, 3.5 to 4. Colour, black, brown, yellow, or red. It is soluble in hydrochloric acid, under separation of sulphuretted hydrogen, and when roasted and heated in the reducing flame, coats the charcoal with oxide of zinc. Its solution in acid gives a white precipitate with caustic ammonia or potash, which is soluble in excess of either of the precipitants.

Section 3. *Hardness under* 7.

Tin Stone.—Sp. gr. 6.4 to 6.9. Hardness, 6 to 6.7. It sometimes occurs almost transparent and colourless, but is generally brownish-black or black. It decrepitates strongly on charcoal, and after a long exposure to the reducing flame, is reduced to metallic tin. It is soluble in acids, only after fusion with an alkali.

Hardness, 7 or above 7.

Quartz.—Sp. gr. 2.6. Hardness, 7. This abundant substance occurs most variously coloured. It forms with soda on charcoal, a clear glass, the solution taking place with effervescence. *Per se,* it is absolutely infusible. It is soluble in no acid but the hydrofluoric.

CHAPTER V.

MINERALS OF ALL THE COMMON METALS; THEIR COMPOSITION AND METHOD OF DETERMINING THEIR CONTENTS BY HUMID ANALYSIS.

MINERALS OF IRON.

Iron Pyrites, Sulphuret of Iron.

Composition:—Iron	. . .	45.74
Sulphur	. . .	54.26
		100.00*

Localities.—This substance is most extensively diffused in all countries. It occurs in large quantities in England, at Alston Moor, Derbyshire, Cornwall, and Wales.

Analysis.†—Treat the ore with *àqua regia* (a mixture of nitric and hydrochloric acids), until all is dissolved but sulphur. Add to the filtered solution, caustic ammonia, a brown precipitate of peroxide of iron falls; the solution also gives a blue with ferrocyanide of potassium, (*iron*).‡ If to another portion of the solution be added chloride of barium, a white precipitate falls,

* Berzelius.

† By this is always understood, *qualitative analysis.*

‡ The word in italics and in brackets, means the substance whose presence is detected by the foregoing tests.

which is insoluble in nitric acid, (*sulphuric acid*), from oxidation of *sulphur* in the mineral, by means of the *aqua regia*. Sulphur remains, as before stated, after the action of the mixed acids; it can be proved to be that substance by its giving off, when burnt, the well known odour of burning sulphur. Hence, the constituents of this mineral are:

Sulphur and
Iron.

Arsenical Iron, or Arsenical Pyrites.

Composition:—Iron	. . .	35.9
Sulphur	. . .	21.4
Arsenic	. . .	42.7
		100.0*

Localities.—It occurs abundantly at Freyberg Muntzic, and other mining districts in Saxony; at Andreasberg, in the Hartz, and in many of the mines in Cornwall.

Analysis.—Act on the ore with *aqua regia*; and if the action be not too long continued, a little sulphur will be left as before, (*sulphur*). Test with ferrocyanide of potassium, a blue precipitate will ensue, (*iron*). The arsenic is best discovered by the garlic odour given off when the mineral is treated on charcoal before the blowpipe.

Specular Iron.

Composition.—This substance is pure peroxide of iron.

Localities.—The largest deposits of this ore are found

* Mitchell.

in the Isle of Elba. It is also found in Saxony, Norway, Siberia, South America, and occurs in very fine crystals in Cornwall.

Analysis.—Its solution may be effected by fusion with carbonate of soda and pulverization of the fused mass, which must then be treated with hydrochloric acid. The solution will give the usual reactions of iron with ammonia, &c.

Hæmatite, Red and Brown.

Composition:—	Peroxide of iron . .	94.6
	Silica	2.8
	Lime	1.2
	Water . . .	1.4
		100.0*

Localities.—It occurs in Bohemia, Saxony, and the Hartz. In Lancashire, and many other parts of England, also in Scotland.

Analysis.—This ore must be treated with carbonate of soda and hydrochloric acid, as the preceding; but the solution in hydrochloric acid must be evaporated to dryness, then moistened with a few drops of hydrochloric acid, and treated with water. The solution must then be decanted or filtered; the silica will remain as a white gritty powder, which if pure, forms a colourless transparent bead by fusion with carbonate of soda, (*silica*). To the filtered solution excess of ammonia must be added, which will precipitate all the iron, which if separated by the filter, will let pass through a colourless liquid, which must be treated with oxalate of

* Mitchell.

ammonia; a white precipitate of oxalate of lime will fall, which is soluble in nitric acid, (*lime*). If a little of the powdered mineral be heated in a bulb tube, water will be given off. From this we conclude, the constituents of this ore are peroxide of iron, lime, silica, and water.

Spathose Iron.

Composition:	Protoxide of iron .	53.0
	Protoxide of manganese	.8
	Magnesia . . .	4.5
	Lime . . .	1.0
	Carbonic acid . .	38.7
	Gangue, (Silica) . .	2.0
		100.0*

Localities.—This ore is very abundant in Styria and Carinthia, also in the Hartz. It is also found in Alston Moor, Cornwall, and Devonshire.

Analysis.—It may be dissolved in nitric acid; the silica will remain intact, and may be examined as before pointed out, (*silica*). A small quantity of solution of hydrochlorate of ammonia may now be added to prevent precipitation of oxide of manganese by means of ammonia, which is now added to precipitate the iron as peroxide, which must be filtered from the liquid containing it. The presence of lime may be ascertained by oxalate of ammonia, which must be added in excess, and the solution again filtered, (*lime*). Magnesia may be detected in the filtered solution by means of phosphate of soda and ammonia, which test will precipitate the granular white precipitate of ammonio-phosphate of magnesia. If the magnesia be present only in small quantity,

* Berthier.

a precipitate will not appear directly, but will, on stirring the solution with a rod, and if only a very small quantity of magnesia (a mere trace, for instance,) be present, the precipitate will appear on the lines made by the rod on the side of the vessel containing the solution. The presence of manganese may be detected by fusing a little of the mineral with soda in a platinum capsule, or by the following excellent test by the humid way, lately proposed by Mr. W. Crum. A small quantity of peroxide of lead must be heated with dilute nitric acid, and the solution suspected to contain manganese added to it; if any be present, an intensely red purple colour will be produced, which will be fully visible when the peroxide in excess has subsided.

Clay Iron Stone may be analysed after this manner; but the solution takes place with more rapidity if it has been previously fused with carbonate of soda. Hydrochloric acid may be employed as the solvent. The presence of carbonic acid in this mineral may be ascertained by its effervescing with an acid.

MINERALS OF LEAD.

Carbonate of Lead.

Composition:—	Carbonic acid . .	13.0
	Protoxide of lead . .	66.0
	Clay	15.3
	Oxide of iron . .	2.3
	Water . . .	2.2
		98.8*

* John.

Localities.—This mineral is found in Scotland at Leadhills and Wanlock Head; in England at Alston Moor, Keswick and Cornwall. It is found in splendid crystals in the mining districts of Saxony.

Analysis.—Pour diluted nitric acid on this mineral in a state of powder: effervescence will take place, owing to the evolution of carbonic acid. Clay will be left. If iodide of potassium be added to the solution, a brilliant yellow precipitate of iodide of lead will take place. Sulphuretted hydrogen produces a dense black precipitate of sulphuret of lead. The presence of iron may be ascertained by means of the ferrocyanide of potassium, and that of water by heating in the mattrass.

Chloride of Lead.

Composition, of a sample from Mendip:—

Chloride of lead .	34.63
Oxide of lead . .	55.82
Carbonate of lead .	7.55
Silica . . .	1.46
Water . . .	.54
	100.00*

Analysis.—This mineral, with the exception of the silica it contains is entirely soluble in dilute nitric acid. The presence of lead can be ascertained as in the former case, as well as the carbonic acid and water. The presence of chlorine is determined by the addition of sulphuric acid. If the mixture be heated, fumes of hydrochloric acid gas are given off; and if a little peroxide of manganese in powder be

* Berzelius.

added to the mixture, free chlorine will be evolved, which, as well as the hydrochloric acid, can be readily detected by the smell.

Sulphate of Lead.

Composition :—Protoxide of lead .	.	72.47
Sulphuric acid .	.	26.09
Water . .	.	.12
Silver . . .	.	.51
		99.19*

Localities.—Sulphate of lead is found in fine crystals at Wanlock Head and Leadhills, at Anglesea and Cornwall, as also at Alston Moor, Siberia, Andalusia and the Hartz.

Analysis.—If this mineral be fused with carbonate of soda it is decomposed; its sulphuric acid will unite with the soda to form sulphate of soda, which is soluble in water, and from its solution sulphuric acid may be precipitated by nitrate of baryta. After the fused mass has been acted on by water it may be treated with acid, and the presence of lead determined by the behaviour of the solution with iodide of potassium and sulphuretted hydrogen.

Sulphuret of Lead.

Composition :—Lead . . .	.	86.55
Sulphur . .	.	13.45
		100.00†

Localities. — Freyberg, in Saxony, and Sala, in Sweden, and Clausthal, in the Hartz. It also exists

* Stromeyer. † Berthier.

in large quantities in Derbyshire, Cumberland, and the northern districts of England.

Analysis.—The ore must be treated with hot dilute nitric acid, and the solution tested for lead with iodide of potassium and sulphuretted hydrogen, and with nitrate of baryta for sulphuric acid, which will be formed by the oxidation of sulphur. Perhaps a little sulphur will remain after the action of the nitric acid, in a free state, its presence can then be readily ascertained by the odour given off when burning. If it contain silica, that will remain after the residual sulphur has been burnt off.

MINERALS OF COPPER.

Sulphuret of Copper, Vitreous Copper.

Composition:—Copper	. . .	76.5
Sulphur	. . .	22.0
Iron		.5
		99.0*

Localities.—This mineral is found principally in Cornwall, Siberia, and Saxony. It is only the first mentioned place that yields it in a crystallized form.

Analysis.—The powdered sulphuret must be treated with boiling aqua regia, until every thing soluble seems taken up; if an excess of ammonia be added to the solution, peroxide of iron will fall down, (this can be readily recognized). The supernatant liquid will appear of an intense azure-blue. If a polished piece of iron be placed in a portion of the original solution, metallic copper will be deposited on it. The liquid may be found to contain sulphuric acid, which is detected

* Klaproth.

by nitrate of baryta. This indicates the presence of sulphur in the mineral. Some free sulphur will doubtless remain after the action of the aqua regia, which can be distinguished, as in the case of the sulphuret of lead.

Grey Copper.

Composition:—	Copper . . .	48.4
	Arsenic . . .	11.5
	Iron	14.2
	Sulphur . . .	21.8
	Silica . . .	5.0
		100.9*

Localities.—Grey copper occurs in Saxony, Hungary, the Hartz and Cornwall; which latter place furnishes the largest known crystals.

Analysis.—Determine the presence of arsenic by means of the blow-pipe. Act on the powdered ore with aqua regia, and filter. Add excess of nitrate of baryta; sulphate of baryta falls, indicative of the presence of sulphur in the ore. If ammonia be now added, arseniates of baryta and iron fall; these were previously held in solution by the excess of acid. Arsenic may be detected in this precipitate by aid of the blow-pipe. The solution is now azure-blue, indicative of the presence of copper, which indication may be confirmed by the plate of clean iron, as in the last experiment. If the precipitate produced by ammonia be dissolved in a little hydrochloric acid, and then treated with ferrocyanide of potassium, a blue colour will be produced, indicative of iron. There will be a residue after the action of the aqua regia on the ore;

* Hemming.

this is a mixture of silica and sulphur; the latter may be burnt off, and the former will remain in a fit state to have its properties tested.

Copper Pyrites.

Composition:—Copper	. .	. 33.12
Iron	. . .	. 30.00
Sulphur	. .	. 36.52
Silica	. . .	. .39
		100.3*

Localities.—This ore is by far the most abundant of all cupreous deposits, and is found in nearly every country. In England, there are immense quantities at Cornwall; at Rammelsburg in the Hartz; at Fahlun in Sweden, and in Saxony, Hungary, Siberia, and America.

Analysis.—This is conducted in the same manner as that of grey copper.

Carbonate of Copper.

Composition:—Oxide of copper	.	. 69.08
Carbonic acid	.	. 25.46
Water	. .	. 5.46
		100.00†

Localities.—It is found near Lyons, near Redruth in Cornwall, and at Alston Moor and Wanlock Head. It is also found in Siberia and Thuringia.

Analysis.—Act on the powdered mineral with hydrochloric acid; effervescence will take place, indicative of the presence of carbonic acid. The solution will give the usual indication of copper with ammonia and

* Rose. † Phillips.

a clean plate of iron. If a little of the ore be heated in a close tube, water will be given off.

Antimonial Copper Ore.

Composition:—Copper	. . .	38.4
Iron	. . . .	1.5
Zinc	. . . .	6.8
Silver	. . . .	.8
Antimony	. . .	25.3
Arsenic	. . .	2.3
Sulphur	. . .	25.0
		100.1*

Localities. — This mineral is principally found at Freyberg, at Kafnik in Hungary, and near Clausthal. It also occurs in Cornwall.

Analysis.—This complex analysis may be completed after the following manner. Treat the pulverized ore with aqua regia as long as any action takes place; filter the solution. To a portion add excess of solution of nitrate of baryta; after neutralizing as nearly as possible with ammonia, a white precipitate will fall consisting of sulphate and arseniate of baryta; arsenic may be detected in this precipitate by the aid of the blow-pipe, and a portion being insoluble in nitric acid will prove the presence of sulphate of baryta, and consequently of sulphur. To the solution filtered from the mixed precipitates of arseniate and sulphate of baryta, add excess of ammonia; the oxides of zinc and copper, first precipitated, will be re-dissolved, but oxide of iron will remain unacted upon; that of copper may

* Rose.

be detected by the deep blue colour the solution takes on the addition of ammonia; if excess of caustic potash be now added to the filtered solution, and the whole boiled, oxide of copper will precipitate, and oxide of zinc be held in solution; if to the filtered solution we now add hydrosulphuret of ammonia, a white precipitate of sulphuret of zinc will fall down. The presence of antimony may be detected by adding an excess of hydrochloric acid to the original solution, and evaporating to a very small bulk, which if poured into water, will determine the precipitation of a white oxichloride of antimony, which may be collected, dissolved in acid, and further tested by the addition of sulphuretted hydrogen, which will induce a precipitate of a deep orange-red colour. The silver in the ore may be detected by acting on a fresh portion of the pulverized mineral with nitric acid, diluting the solution with water, and filtering. If we now add hydrochloric acid, or any soluble chloride to the filtered solution, a white curdy precipitate of chloride of silver will be produced, which is soluble in excess of caustic ammonia.

Plumbiferous Grey Copper.

Composition:—	Copper . .	8.000
	Antimony . .	27.600
	Sulphur . .	25.120
	Iron . . .	2.768
	Lead . .	24.684
	Silica . .	11.828
		100.000*

* Mitchell.

This mineral must be pulverized and acted upon by aqua regia, as in the last case, and the analysis proceeded with in exactly the same manner. The lead in this mineral may be detected by the addition of sulphuric acid, or a soluble sulphate to the solution, when a white precipitate of sulphate of lead will fall down, which is characterized by its solubility in caustic potash, and furnishing a button of metallic lead when treated on charcoal before the blow-pipe.

MINERALS OF ZINC.

Sulphuret of Zinc, Blende.

Composition:—

Zinc	61.1
Iron	3.8
Sulphur . . .	35.1
	100.0*

Localities.—It occurs in England, principally in Derbyshire, Cumberland and Cornwall. It also occurs in Hungary, Transylvania and the Hartz.

Analysis.—Digest the powdered ore in nitric acid or aqua regia; filter the solution; add to a portion of it nitrate of baryta, a white precipitate will fall, indicative of sulphur having existed in the ore; to another portion of the solution add ammonia, a brownish white precipitate will fall, consisting of a mixture of oxide of zinc and peroxide of iron; on the addition of an excess of ammonia, the oxide of zinc will be dissolved; the oxide of iron may then be separated from it by the filter, and the presence of zinc determined in the solution by saturating

* Mitchell.

the excess of ammonia with an acid, oxide of zinc will again fall, and its identity may be proved by moistening a small portion with nitrate of cobalt and igniting before the blow-pipe, when it will acquire its characteristic green tint. The presence of iron in the solution may also be detected by the ferrocyanide of potassium, producing the well-known Prussian blue.

Carbonate of Zinc, Calamine.

Composition :—Oxide of zinc . .	64.8
Carbonic acid . .	35.2
	100.0*

Localities.—It is found at Matlock, Mendip, and Wanlock Head. It is also found in Hungary, Silesia, the United States and in Carinthia.

Analysis.—Treat the powdered ore with hydrochloric acid; a gas will be given off (with effervescence), which is inodorous; hence it is carbonic acid. Zinc may be detected in the solution, as in the preceding case.

These few examples of qualitative analysis will, it is hoped, be a sufficient guide to the student in ascertaining the presence of the most common metals by the humid method. Neither space nor the original plan of the work will allow the author to enter more fully into this particular; and for the metals not here mentioned, reference must be made to some work especially devoted to analysis; for instance, Rose's Analytical Chemistry, or Parnell's Elements of Chemical Analysis.

* Smithson.

CHAPTER VI.

THE ASSAY OF IRON.

BERTHIER divides all substances containing iron, which are ordinarily assayed, into five classes, as follows:

Class 1.—Pure ferruginous substances, as the Hæmatites, &c.

Class 2.—Iron ores mixed with quartz, but containing no other substances.

Class 3.—Ferruginous matters, containing quartz and various bases, but no lime.

Class 4.—Ferruginous substances containing one or many bases, as lime, magnesia, alumina, &c.

Class 5.—Iron ores, containing silica, lime, and another base, which ores are fusible, *per se*.

The following are Berthier's directions for operating in the ordinary manner.

"In the assay of iron ores, naked crucibles, either of clay or black-lead, or crucibles lined with charcoal, are employed.

"The button of metal does not adhere to naked pots, but the slag adheres very strongly; so much so, that it cannot be detached with any degree of accuracy for weighing, (which in some of M. Berthier's processes is of importance). Black-lead pots allow neither the slag

or button to adhere, but the former dissolves much argillaceous matter from the pot, so that its weight is much increased, and the assay cannot be verified. In naked crucibles charcoal is always obliged to be added to the assay, to reduce the oxide of iron; in which case, if an excess be added, it prevents the button from completely forming, so that globules remain in the slag, (I have found, however, that this may be avoided). Neither do naked crucibles resist the fire as well as those lined with charcoal; because the lining supports the sides when they soften. The charcoal lining also allows us to finish the assay without adding any re-agent to the ore; the button can be readily taken out, because it does not adhere to the charcoal; and lastly, the earthy matters in the ore, which have formed a slag, may be collected and weighed; or if we have added any flux to the ore, the total weight can also be ascertained.

"After having finely powdered and sifted the iron ore, a determinate weight must be taken (I find 200 grains to be the most convenient quantity in ordinary cases); a certain weight of the requisite flux must be well mixed with the ore in a mortar, and the whole placed in the crucible, (which must be lined with charcoal), in which it must be firmly pressed by a pestle or other appropriate instrument. The crucible must then be filled with successive layers of charcoal powder, slightly moistened with water; a cover is then luted on it, and the whole placed in the fire. The fire is allowed to burn gently for about an hour, and the heat is then raised to whiteness for about an hour, or an hour and a

quarter; the crucibles are then taken out, allowed to cool, broken, and the button and flux removed.

"The whole fused mass must be weighed, and then the button of metal must be carefully separated from the flux, and weighed; sometimes small globules of iron are found adhering to the flux, in which case, they must be removed, and added to the button before weighing. Even when very small, their removal can be readily effected; the flux must be finely pulverized, and placed on a sheet of paper; a magnet must then be drawn gently over its surface, which method of procedure will ensure the separation of all metallic particles. If the weight of metal obtained be now deducted from the weight obtained in the first weighing, the difference will be the weight of flux.

"Good buttons of metal, when wrapped in pieces of thin tin-plate, and struck with a heavy hammer, on the anvil, flatten slightly before they break; they ought to be grey or greyish-white, and the grain fine, or tolerably fine. Bad buttons break readily, without changing form, some even pulverize; they are generally very white and crystalline on the surface."

METHOD OF CONDUCTING THE ANALYTICAL ASSAY.

By the method of assay just pointed out, we have no means of ascertaining whether the per centage of metal obtained is exact or not; and the accuracy of the result cannot be reckoned on without a second assay. But by performing certain simple operations on the mineral before fusion, a double assay may be dispensed with, and much useful knowledge gained as to the nature of

the body; indeed, this method is only second to an analysis by the humid method. These operations are comprised in roasting or calcining, to drive off any volatile or combustible matters, in treating the ore with certain acids, the object of which is to ascertain the amount of insoluble matter, by difference of weight, before and after the action has taken place.

The hydrated ores are calcined to estimate water; those ores containing manganese, to reduce it to a fixed and known state of oxidation (sesquioxide). The carbonates are roasted to expel carbonic acid, and the ores from the coal formations to burn the combustible matter with which they are mixed.

Slags and dross are also roasted to free them from charcoal, with which they are mixed. A simple calcination sometimes is sufficient, as in the case of the carbonates; but where mixtures of per and protoxide of iron are to be assayed, they must be subjected to a long roasting, in order to convert all the contained protoxide into peroxide.

Diluted and cold nitric and acetic acids are employed for minerals whose matrix is purely calcareous or magnesian, as these acids dissolve the earthy carbonates, without attacking either stones, clays, or the oxides of iron. The residue is to be well washed, dried, and weighed, and the amount of carbonates calculated by the difference. It is now to be treated with boiling hydrochloric acid, or what is preferable, by *aqua regia*. The ores which contain substances insoluble in these acids, are generally of a clayey or flinty nature. These are to be weighed, and according to their weight, that

of the flux to be added in the assay is determined, as will be shown hereafter.

It must be borne in mind, however, that the clays are not absolutely insoluble in hydrochloric acid, for a certain quantity of alumina is always dissolved, which is greater in proportion to the proportion existing in the clay.

The ores containing titanium are boiled with concentrated sulphuric acid, after they have been reduced to the finest possible state of division. All the oxides of iron, titanium, and manganese are dissolved, and the stony gangues which resist the action of this acid can be estimated. The utility of this estimation will be pointed out as we proceed.

When all the operations necessary for each particular case have been completed, we know the proportion of volatile substances, of substances soluble in acetic acid, and those insoluble in hydrochloric and sulphuric acids, contained in the substance under assay.

The suitable flux is then added, and the fusion proceeded with as usual. In general we have a choice of a variety of fluxes; but if the assay is to be verified and made as accurately as possible, fixed fluxes must be employed, or fluxes which lose only a determinate amount of volatile matter. Carbonate of lime and carbonate of magnesia, are examples of this class of flux.

Let A be the weight of the rough or non-calcined ore. B the weight of the same calcined. C the weight of the fluxes in a rough state. D the weight of the same calcined. P the weight of matter insoluble in

hydrochloric or sulphuric acids. R the weight of the fixed substances soluble in acetic or nitric acids: a weight which can be readily calculated when we know the loss which the ore, not treated by acids, suffers by calcination; and the residue of the treatment of this substance by acetic or nitric acid. M the weight of the button of metal and scattered globules. S the weight of the slag, and O the loss of weight in the assay which represents the quantity of oxygen disengaged during the reduction.

The following is the disposition of the data from which, at one view, all the useful results of the assay can be determined.

In the assay has been employed:

A, rough ore = calcined ore . . .	B
C, of rough fluxes added = fixed flux	D
Total of fixed matter	B + D

The result has been:

Metal — M, Slag — S } Total . . .	M + S
Loss	O

Fluxes added	D
Vitrifiable matters	S — D
Substances insoluble in hydrochloric acid, &c.	T
Substances soluble in hydrochloric acid, &c.	S—D—T
Substances soluble in acetic acid . .	R
Substances insoluble in acetic acid, and soluble in hydrochloric acid. .	S—D—T—R

When the iron in the substance assayed is in a known degree of oxidation, and when but little manganese is present, the quantity of oxygen O, ought to correspond very nearly with the quantity of metal M produced; if it does, the assay must be correct.

A rigorous correspondence between the two numbers, however, cannot always be obtained, because the iron is not pure, but always contains carbon, so that in ordinary assays the peroxide of iron loses but from twenty-nine to thirty per cwt. of oxygen.

On the other hand, the quantity of iron remaining in the slag makes up in part for the carbon combined with the metal produced; but when the assay has been made with a suitable flux, the quantity of oxide remaining is very small, and never exceeds one per cwt. of the weight of the slag. When the iron is in an unknown degree of oxidation the loss O produced in the assay gives the degree, if it has been made without accident; but if there is any doubt, and the result is of importance, the assay must be recommenced for verification. If the ferruginous matter contain manganese, and if that metal be in the state of protoxide, the verification just described can be made without modification, because the manganese dissolved in the slag is always at the *minimum* of oxidation; and when a sufficient quantity of flux is employed, the amount reduced is of no consequence. But when the manganese is in the state of red oxide, it parts with a certain quantity of oxygen on being reduced to the *minimum* of oxidation, and which quantity is estimated in the loss O, so that a perfectly accurate verification cannot be made. Nevertheless, the difference between the loss

O, and the quantity of oxygen calculated from the metal M, cannot be very great, because the red oxide of manganese loses but .068 of oxygen in its transformation to protoxide.

Titanic acid behaves in iron assays exactly as the oxides of manganese; it disengages at most but .06 of oxygen when dissolved in the earthy glasses in contact with charcoal.

It sometimes happens that the assay is not fused, or but imperfectly so. This can happen from two causes; first, because the heat has not been sufficiently strong or continued; secondly, because the flux has not been employed in proper proportion, or has not been calculated to form fusible compounds with the foreign matters mixed with the oxide of iron. In both cases the oxide of iron is completely reduced; and if the assay has been made with care, the loss of oxygen indicates the amount of iron in a very approximative manner, and nearly always with an exactitude which is surprising to those not accustomed to this kind of operation.

The assay buttons which are not fused have a grey and homogeneous appearance. They flatten under the hammer. Take a metallic lustre by friction with a file, and disengage hydrogen on being moistened with hydrochloric acid. The iron they contain is in imperceptible particles.

In the imperfectly fused buttons the iron is disseminated in globules throughout the whole mass of slag, or form a scoriform button mixed with much slag, without the possibility of complete separation.

Sometimes there is not even an agglomeration, and the mixture submitted to assay forms but a greyish

metallic powder, in which case the assay is useless, as it is impossible to collect the whole without loss, even by washing the charcoal lining with the greatest care.

This method of assay, says Berthier, cannot be too much recommended to iron masters, for all the facts necessary to be gotten, in relation to the ores they smelt, are imparted in a very simple and expeditious manner; and by varying the experiment, and by employing as fluxes the ordinary substances used for that purpose, a knowledge of the mixtures which will answer best in the high furnace, may be obtained without loss of metal or much expence.

The Assay of substances of the First Class

May be effected in charcoal lined crucibles without the addition of any flux, because the foreign matters found in ores of this class are generally fusible *per se*; nevertheless it is always better to add a flux; first, because there is no risk incurred of being obliged to recommence the assay; and, secondly, because a certain amount of slag is always very useful, by enabling the metal to form into one single button. Borax may be employed for this purpose; but it is better to use some very fusible earthy silicate.

Assay of Ores of the Second Class.

In order to render fusible the silicious matters constituting this class, carbonate of soda may be employed, a mixture of chalk and alumina or clay; or a mixture of carbonate of lime and dolomite. (*Magnesian limestone*).

Assay of Ores of the Third Class.

The ores belonging to this class are the most common of all. They all fuse exceedingly well by the addition of carbonate of lime, amounting to about half or three-fourths of the weight of gangrene, and mixed with the oxide of iron. The ores of this class may also be assayed by the addition of carbonate of soda.

We shall here give the result of an actual assay made after the manner described at page 229.

The assay of an argillaceous iron ore.

Loss by calcination and roasting .		. 33 per cent.
Insoluble in hydrochloric acid .		. 14.2 do. of clay
100 grs. of the crude ore, give of calcined ore		. 67 grs.
10 grs. of carbonate of lime = lime		. 5.6
Total . .		. 72.6
Amount of metal .	36.3 grs.	= 57.0
Do. slag .	20.7 grs.	
Oxygen . .		. 15.6
Flux added . . .	5.6 grs.	
Vitrifiable matter .	15.1 grs.	
Insoluble matters .	14.2 grs.	
Soluble matters . .	.9 grs.	

Assay of Substances of the Fourth Class.

In the assay of those ferruginous matters which cannot fuse because there is a deficiency of silica, a certain amount of silicious sand must be added; but it is nearly always necessary to add at the same time

either lime or some other base or even two bases at once.

The spathose iron ores containing much manganese fuse with the addition of quartz alone; on the contrary, those which contain much magnesia require besides quartz a certain amount of lime.

When the spathose irons are mixed with stony gangues, the proportion of the latter is determined by treatment with aqua regia, and as these gangues are nearly always quartzose, besides the suitable amount of silica which must be added, an amount of lime equal or nearly equal in weight to the whole amount of silicious matter contained in the ore must be superadded.

For aluminous minerals a mixture of silica and lime is necessary. Although silica and lime alone may be employed in the assay of titaniferous minerals, it is always better to add a small quantity of alumina or magnesia, as such an addition renders the slag more fusible. Lastly, very calcareous ores are often rendered fusible by the mere addition of silica; because they contain nearly always a certain amount of alumina or another base; but it is always better to add, as a flux, a very silicious white clay.

Iron Ores of the Fifth Class.

These merely require fusion in a charcoal crucible, without the addition of any flux.

The method of assay I pursue in my own laboratory is readier of execution than that just described, and I find it applicable to all kinds of ores, at least all those kinds which generally fall to the lot of the ironmaster or assayer to examine.

The furnace I employ is the ordinary wind furnace, capable of producing a full white heat; the crucibles, those termed "London pots," which are decidedly more refractory than either the Cornish or the Hessian; indeed, pieces of either of the last named crucibles can be softened in a London pot, without any alteration in the shape of the latter. I do not employ the crucibles lined with charcoal, but in a naked state.

The treatment of the ore is as follows: very finely pulverize the sample to be assayed, and weigh out 200 grs., which mix in a mortar with 100 grs. of quick-lime, and from 20 to 40 grs. of charcoal, in proportion to the apparent richness of the ore: it is always advisable to have a slight excess of charcoal. When well mixed, place in a crucible, and cover over with about 300 grs. of powdered bottle glass:—taking especial care that it contains no lead. Two crucibles must be prepared in this manner, and both placed in the furnace, when it is at a dull red heat. Allow the heat gradually to increase in intensity, until it arrives at a very bright red, or yellowish white heat, which ought to be in about three quarters of an hour, then increase the heat to whiteness for half an hour, and take the pots out gently; tap their bottoms on a brick, in order that the fluid metal may collect into a single button, and allow them to cool gradually. When perfectly cold break them; and if the assay has been well conducted, the two buttons will not differ $\frac{1}{8}$th of a per cent. from each other, and the result is certain to be accurate. This (the double assay) is the only method of verification, I find it

necessary to adopt. Some advise the addition of borax, but I do not find it useful; it is, in fact, rather injurious, as it alters somewhat the character of the obtained metal. If the nature of the gangues, &c., be wished to be ascertained, the humid method of analysis must be had recourse to after the manner described below.

Humid analysis.—Some iron ores dissolve very readily in hydrochloric acid, or aqua regia, others do not, even when they are in a very fine state of division; but all do readily after fusion with an alkali, or alkaline carbonate, as of potash, or soda: hence it is advisable to fuse the finely pulverized ore with alkali previous to attempting its solution in an acid.

The ordinary constituents of clay ironstone, (which is about the most complex) are: oxide of iron, carbonic acid, lime, silica, alumina, and oxide of manganese.

Iron is always estimated in the state of calcined peroxide, as it is the only compound of iron which has a fixity of composition, rendering it applicable for an accurate analysis. If, however, during its calcination, a portion is reduced by any carbonaceous matter, it may be ascertained by its black colour, and by being attracted by the magnet. In order to reduce it to the state of peroxide, it is only necessary to add a few drops of nitric acid, and calcine anew.

It is necessary also to bear in mind, that organic substances prevent the precipitation of peroxide of iron by the alkalies.

Caustic ammonia is employed in preference to potash or soda in the precipitation of peroxide of iron.

The following is the method of analysis: the ore must be reduced to fine powder, (100 grs. is a good quantity), and well mixed with five or six times its weight of carbonate of potash or soda, and heated to full redness for one hour in a platinum crucible. When cold, the crucible is placed in a large evaporating basin, with a little water, pure hydrochloric acid is then poured upon it, the mass dissolves with effervescence, and more acid must be gradually added, until no more action seems to take place. The solution being finished, the crucible washed with distilled water, and the whole solution, with the washings, evaporated to dryness. Towards the end of the operation, the partially dried mass must be continually stirred, in order to prevent losses by the spirting, which will otherwise take place. The contents of the basin are then moistened with hydrochloric acid, and the whole left to itself for about an hour.

It is then mixed with a small quantity of distilled water, and thrown upon a filter; all but the silica will have been dissolved, and pass through the filter. The silica remaining on the filter, is to be well washed with hot water, dried, ignited in a platinum crucible, and weighed.

The filtered liquid must have an excess of caustic ammonia added to it. Alumina, peroxide of iron, (and if the ore contained manganese), oxide of manganese will be thrown down. These solid matters must be separated from the liquid by filtration, and after being well washed, acted on by boiling caustic potash, which will dissolve the alumina, and leave untouched the oxides of iron and manganese. The

alumina may be separated from its solution in caustic potash, by neutralization with hydrochloric acid, and the addition of a slight excess of ammonia. The whole must then be thrown upon a filter, and the remaining alumina well washed with hot water, dried, ignited, and weighed.

The following seems to be the readiest method of separating the oxides of iron and manganese. They are to be dissolved in as small a quantity as possible of hydrochloric acid; the iron existing in the solution in the state of peroxide, and the manganese as protoxide. This may readily be effected by boiling the liquid with a few drops of nitric acid, which will peroxidize the iron, and in case it should have had any such effect on the manganese, add a little sugar, and boil anew, in which case the manganese will alone be deoxidated. The solution is then to be exactly neutralized, with solution of carbonate of ammonia added drop by drop; the liquid being, at the same time, continually agitated. After a short time the solution becomes cloudy, and hydrated peroxide of iron deposits: it may be made to precipitate immediately by raising the temperature of the solution to the boiling point. The solution must then be filtered from the deposited peroxide of iron, which must be ignited and weighed; every 40 parts of which, correspond to 28 parts of metallic iron. The filtered liquid contains the manganese, which may be obtained as oxide by evaporating to dryness, and igniting. Every 100 parts corresponds to 93 parts of the protoxide of manganese.

The liquid from which the mixed oxides of iron,

manganese, and alumina, were separated, is to be very carefully neutralized with nitric acid, (taking care, however, to have no excess; it being better that the solution should be rather alkaline than otherwise), and the lime it contains precipitated with oxalate of ammonia. The oxalate of lime so formed, must be separated by filtration, washed, ignited, and when cold, moistened with concentrated sulphuric acid, and again ignited, and weighed as sulphate of lime; every 68 parts of which correspond to 28 parts of quick-lime, or 50 of chalk.

The solution just freed from the lime, is to be rendered ammoniacal by means of ammonia, and phosphate of soda added to it, which will determine the precipitation of the magnesia it contains, in the state of the ammonio-magnesian phosphate, which must be washed, dried, ignited, and weighed. Every 100 parts correspond to 36.67 parts of magnesia.

CHAPTER VII.

ASSAY OF COPPER ORES.

In the assay of copper ores by the dry way, all minerals containing that metal are divided into four classes.

Class 1.—Comprises substances containing neither sulphur, arsenic, or any foreign metals but iron.

Class 2.—Comprises ores, &c., containing sulphur, but no other metal than iron.

Class 3.—Comprising the sulphurets which contain other metals than iron, as arsenic, antimony, lead, &c.

Class 4.—Various alloys.

Assay of the substances contained in the First Class.

Rich Ores.—The assay of the ores of this class is comparatively facile, and the results obtained are very exact.

The assay is conducted by the aid of black flux, as follows: The powdered ore must be well mixed with about 3 parts of ordinary black flux, and placed in a crucible, (which it ought not to fill more than two-thirds), and covered with a thin layer of black flux, and heated gradually in an ordinary furnace, for about a quarter of an hour; a cover is then placed on the pot, and the heat increased to bright redness, which will be sufficient to melt the copper. A quarter of an hour, at the full heat, is ample, and no fear need be entertained

that metal will be lost by excess of heat, for copper is perfectly fixed at the above temperature.

When the crucible is cold, it must be broken; and if the assay has succeeded, a button of copper will be obtained, which ought not to adhere to the crucible, and which separates readily from the slag.

Poor Ores, &c.—The slags produced by the treatment of copper ores in the large way, can be assayed after the plan just described, when they are rich; but when they contain no more than about 2 per cent. of copper, that method will not succeed; because, the small quantity of copper they contain would remain in the flux, either combined or disseminated in very small grains. In this case, if the slag be attackable by acids, the assay must be made by the wet way; if they be unacted on, or nearly so, by acids, their assay must be conducted in a charcoal crucible, in the same manner as iron, and at the same temperature. Sometimes it may be made without the addition of any flux; nevertheless, it is always better to add 15 to 20 per cent. of chalk.

Those which are acted on by acids generally contain much oxide of iron, and assimilate very much in their character to finery slags.

In case the dry assay is made as above stated, the copper will be found alloyed with much iron, from which it must be separated in the wet way.

To estimate the per centage of copper by the humid method, the substance must be finely pulverized and acted on by boiling *aqua regia*, (made in the proportion of 3 parts hydrochloric acid, and 1 part nitric acid); or still better by hydrochloric acid alone, if it be attackable by that agent. After all action has ceased, evaporate

nearly to dryness; moisten the residue with hydrochloric acid, dilute with water, and filter, in order to separate silica and other insoluble substances.

The copper has now to be separated by precipitation, which is most readily effected by placing in the liquid a well cleansed plate of iron, and leaving it there until all the copper is thrown down.

This precipitation takes place either when the solution is hot or cold; in the latter case, it is requisite to leave it for at least twenty-four hours; in the former more especially; if a boiling heat be given, it takes place at most in an hour or two. It must be borne in mind, however, that in both cases the solution must be kept decidedly acid, otherwise an insoluble sub-salt of iron will be thrown down with the copper, and much interfere with the exactness of the result. Either hydrochloric or dilute sulphuric acid will serve for the acidification of the liquid.

When all the precautions just pointed out have been taken, the precipitated copper is in thin light scales, of a fine red colour, and perfectly pure. It must be washed rapidly in a large quantity of water, until the last washing comes off perfectly tasteless, then dried at a very gentle heat (212° F.) and weighed. If it be dried at a stronger heat, the finely divided metal has a great tendency to combine with oxygen and form an oxide, which of course would be much heavier than its contained metal; therefore the result would be a greater per centage than the substance actually contained.

In case the greatest possible exactitude be wished for, it is better to precipitate the copper by means of sulphuretted hydrogen than iron. This re-agent detects

the smallest traces, and hence it is desirable to employ it when very poor substances are to be examined. It is better, when this gas is employed, to dissolve the ore or slag in hydrochloric acid alone, if possible, and the solution must contain an excess of acid.

The precipitate must be collected, washed, and dried. It must then be perfectly roasted in a small platinum or porcelain capsule, and weighed as deutoxide of copper, every 40 parts of which correspond to 32 parts of pure copper.

Assay of ores, &c. of the Second Class.

The substances belonging to this class are either sulphurets or sulphates.

Sulphates.—The sulphates of copper give very pure copper with a reducing flux, and a slag containing a double sulphuret of the alkali and copper. The larger the proportion of flux employed, so is the quantity of sulphuret produced, and a correspondingly small quantity of metallic copper reduced. But if the sulphate of copper be fused with an alkaline flux, which contains only the exact quantity of the reducing agent, to reduce its contained oxide of copper, then all the metal is reduced, and the slag contains only sulphate and no sulphuret.

Thus, it has been ascertained, that the neutral anhydrous sulphate of copper gives: firstly, 27 per cent. of copper with 3 parts of black flux (about two-thirds only of that which it contains), and a black crystalline slag, containing much sulphur. Secondly, with 2 parts of the same flux, 37 per cent. of copper, and a clean grey slag, containing very little sulphur. When but one

part of black flux is employed, there is not a sufficiency of carbon to reduce the whole of the oxide, and only about 12 to 14 per cent. of metallic copper is produced, which is enveloped in a red, vitreous, opaque slag, composed of protoxide of copper and alkali, and above which, is a layer of fused sulphate of potash, which is crystalline and colourless.

It may thus be seen, that in order to extract the whole of the copper from a sulphate in this manner, that the proportion of reducing flux which will give the maximum result, and which also will produce a slag containing but little sulphur, must be arrived at by guess-work.

The sulphates of copper, however, are completely decomposed by heat; and the easiest and best method of assaying them, is to expose them to a white heat in a platinum crucible until nothing more is given off. The residue is oxide, which can be fused with three parts of black flux, when all the copper contained in it is exactly and readily separated in the metallic state.

The compounds of oxide of copper with sulphuric acid may be assayed by fusion in an earthen crucible with from 1 to 2 parts of carbonate of soda; the fused matter must be poured into an ingot-mould, pulverized, and re-fused in the same crucible, after mixture with its own weight of black flux. By fusion with carbonate of soda, the sulphate of copper is decomposed, and sulphate of soda and a compound of the oxide of copper and alkali formed, which is afterwards completely reduced by black flux, without the possibility of the formation of a sulphuret of copper.

Sulphurets.—Copper, combined with sulphur, is

assayed both for regulus, or matt, and metal. The assay for regulus has for its purpose the determination of the relative quantity of sulphuret and gangue in the ore; and, in consequence, the quantity of regulus a given quantity of ore would produce in the large way.

The assay for regulus is simple and easy. It consists in acting upon the ore with a substance capable of determining the fusion of the gangue, but exercising no decomposing action on the sulphurets. No flux fulfils these conditions better than borax. With equal parts of glass of borax, all ores to be assayed in this manner, fuse very readily at a full red heat. Naked pots are generally employed for this kind of assay; but then great precaution must be taken to collect the substance without loss; because, in the first place, it may adhere to the crucible, and because it is exceedingly brittle. This inconvenience may be avoided by the employment of crucibles lined with charcoal, which Berthier says are indispensable in the assay of very poor ores. In my own experience, I have never found any inconvenience from the use of naked crucibles, where the proper degree of heat has been employed.

If it be thought necessary to use as fluxes the same substances as those employed in the large way as lime, baryta or quartz, the assay must be made at the same temperature as an iron assay in a lined crucible —a plain one will answer every purpose.

It is essential to remark that every regulus produced is composed of sulphurets, containing the minimum amount of sulphur; since all the sulphurets contained in ores are found at a higher degree of sulphuration, a certain quantity of sulphur sublimes during fusion, so

that the amount of gangue cannot be deduced from the quantity of regulus produced.

By the humid method, however, the proportion of gangue can be readily determined, and in consequence that of the sulphuret; but the proportion of regulus producible by fusion with borax cannot be determined by this experiment, because the nature of the sulphurets being unknown, the amount of sulphur they would give off during fusion could not be found. By a combination of the two methods a complete knowledge of the nature of the substances assayed is obtained.

In order to make the assay by the humid method, the substance is pulverized and acted on by acetic acid which dissolves but the calcareous gangue. It is then dried and weighed. The residue is then boiled in aqua regia, and washed, and roasted, to burn off sulphur. The stony gangues left, after this process, are weighed, and the metallic part calculated by difference.

The process just described supposes the substance assayed does not contain a notable proportion of oxide of copper or iron; in case it did they would be dissolved by either acid, and the true proportion of the sulphurets could not be ascertained. This proportion also could not be determined by the dry way; but the proportion of regulus producible on the large scale, could be very accurately determined by fusing the ore with borax or any other non-desulphurating flux; and that is all which is generally required.

The assay of the ores of copper containing sulphur, requires necessarily a roasting; and a roasting made with such care that neither sulphur nor sulphuric acid shall remain in the substance; without which in the

subsequent fusion with a reducing flux the slag would retain traces of copper.

Berthier says, all other processes which have been attempted to be employed have been without success, and present more difficulties than the usual method of manipulation.

The presence of iron whose sulphuret has so great an affinity for that of copper, presents an especial difficulty which it appears exceedingly hard to overcome.

With nitre all the copper can be extracted (from the sulphuret), but with difficulty, and finding by repeated experiments the quantity which produces the maximum result. Sulphuret of copper fused with a mixture of an alkaline carbonate and metallic iron, allows a certain quantity of copper to be set free; but this quantity never surpasses above three-fourths of that contained. This maximum is obtained by using four parts of carbonate of soda, and thirty to forty per cent. of iron filings. The slag is black and homogeneous.

Copper pyrites is decomposed by the alkaline carbonates, but without the production of metallic copper. A black crystalline homogeneous slag is formed, which contains an alkaline sulphuret, a sulphuret of iron, sulphuret of copper, and oxide of iron.

With black flux a very fluid homogeneous crystalline slag is produced, the colour of which is black with a metallic lustre, and in which not the smallest trace of metallic copper can be distinguished. The result is the same as the addition of iron, which is perfectly inert, and remains disseminated in the slag.

Copper pyrites is readily attacked by nitre, and either

copper or sulphuret of copper can be separated from it by means of this reagent. But in order to arrive at this result, it is necessary to guess the quantity of nitre; so that it is evidently not a good method of assay.

It is necessary, in order that the slag be fluid, to add a little borax or an alkaline carbonate; so that the reduced metal may collect into one button. With one part of nitre, two of carbonate of soda, and one of borax, pure copper pyrites gives from thirty-six to forty-six per cent. of sulphuret; with double that quantity of nitre it gives thirty per cent. of metallic copper.

The roasting of pure sulphuret of copper, or copper pyrites, (a mixture of the sulphurets of copper and iron), is a very easy operation—so Berthier observes. The only precaution necessary is to manage the heat very carefully for some time, in order to avoid agglomeration; but after a certain quantity of oxide has been produced, a red heat may be employed without inconvenience. The ore must be stirred continually so as to bring necessarily every portion into contact with the atmosphere.

When the roasting is considerably advanced, it is best to push the heat to bright redness from time to time, in order that the sulphates and sulphurets may mutually decompose each other. Lastly, when no more sulphurous acid is evolved, the heat may be raised to whiteness for some minutes to decompose the last traces of sulphates—which decomposition this process very seldom effects. When the roasting has been executed with care, the substance fused with three, or four times its weight of black flux, gives nearly all the

copper it contains, and all the contained iron remains in the slag, partly as oxide and partly as metallic iron; but in a very finely divided state. If the roasting has been imperfect, the slag will contain sulphur, which will abstract a considerable amount of metallic copper from the assay button.

When the ore is poor and contains much iron, the slags resulting from the assay are always pasty; and some care must be taken, otherwise they will retain as much as two to three per cent. of copper.

It is preferable, in these cases, to estimate the copper by the wet way by dissolving in aqua regia, and subsequent precipitation by metallic iron. The humid method (Berthier) of assay is preferable for the ores belonging to this class.

Mr. Lewis Thompson has proposed several methods for the assay of copper ores, both by the wet and dry method. The following is an outline of his wet and dry process.

In the dry process, the ore is pulverized finely and heated to redness in a crucible, and nitre thrown upon it till there is an excess which may be determined by the deflagration producible by an excess of that agent, on the introduction of a piece of charcoal. When this happens, charcoal is to be added until all the excess of nitre is destroyed. The pot and its contents is then to be raised to a full red heat for about a quarter of an hour, and the button produced in the operation weighed.

This process is not perfect, for reasons before pointed out. And the amount of copper obtainable from the same sample of ore varies in its quality very much.

The same, however, cannot be said of his humid

process; which is, indeed, as perfect as it is possible to suppose a process. In the ordinarily known humid method, the ore is treated most commonly by nitric or nitro-hydrochloric acid, in which case the excess of nitric acid has to be got rid of before the precipitation can be effected. It is either done by evaporation to dryness and moistening with hydrochloric acid, or by an admixture with sulphuric acid, and boiling to expel the excess of nitric acid; both of which are troublesome processes. In Mr. Thompson's method all these are avoided, and the operation much shortened. There is, however, a drawback to the general employment of it, and that is the disagreeable suffocating and extremely deleterious fumes of euchlorine which are disengaged; and unless the operation be carried on under a hood with a good draught, fatal consequences may ensue, when the operator has tender lungs. The process is as follows:

Pulverize finely 100 grs. of the ore, and pour upon it about two ounces of concentrated hydrochloric acid, and add some chlorate of potash (about as much as will cover a sixpence), and stir well together with a glass rod. After all action has ceased add more chlorate, if that added previously has disappeared; if not place the basin containing the mixture on a sand bulk, and keep adding chlorate until all action ceases. Then add to the solution some water, allow the undissolved matter to subside, and decant the clear liquid. The residue is generally composed of gangue, free sulphur, and unacted on ore. It is to be carefully dried and roasted, to burn off sulphur, &c., and then treated with acid and chlorate as before mixed with water, and the clear solution de-

canted; if the residue be not perfectly white, roast again; attack with acid, &c., until it is. The mixed solutions are now to be filtered, a strip of clear iron placed in, and the precipitate of the copper, &c., is to be made as before described.

Mr. Maughan contrived a very clever, but rather expensive process, of assaying copper ores, by placing the powdered substance in a porcelain tray in a red hot tube, and passing oxygen gas over the ignited mass. The ore was thus rapidly roasted (but with the same objection to the ordinary and imperfect method of roasting, to which I shall presently advert); it was then mixed with black flux, and run down, and a button of metallic copper obtained. I reduced the expense of this process much, by substituting common air for pure oxygen. I found it answer as well; but it was still far from a perfect process.

The following is the method I now employ, and which I have been led to adopt from an investigation connected with the theory of roasting copper ores, conducted by my friend, Mr. Field, and self: inasmuch as we have found it next to impossible to completely rid the ore of sulphur by the ordinary method of roasting.

Two hundred grains of the ore are to be finely pulverized, and roasted as completely as possible with all the precautions before pointed out; about 20 grains of finely powdered carbonate of ammonia are to be very gradually added to the roasted mass, while still red hot, stirring carefully and assiduously, until all be well mingled; the whole is now to be submitted

to a strong red heat for a few minutes, after which not the slightest trace of sulphur can be detected in the roasted ore by the most delicate chemical tests; it is, indeed, but a mixture of oxides of copper and iron, with gangue. The reason carbonate of ammonia is employed, depends upon the volatility of sulphuric acid in its vapour, and it is the last portion of sulphur existing in the state of sulphuric acid in the roasted mass, which is so difficult to get rid of, unless the above process be employed; and it is that which, if allowed to remain, will mar the otherwise carefully conducted assay.

After the roasting is fully complete, mix very carefully in a mortar the contents of the crucible, with 50 grs. of lime, and from 20 to 40 grs. of charcoal, (according to the richness of the ore), together with 300 grs. of dry carbonate of soda; place the whole in the same crucible in which the roasting was conducted, and place on the top of the whole 200 grs. of borax. In this condition, put the pot in the fire when it is at a dull red heat, and allow it to remain so for about a quarter of an hour, and then push the heat to bright redness for about the same time, or until the flux has a perfectly smooth surface. Allow the crucible and its contents to cool thoroughly, and then break it, when a button of copper will be obtained, which will correspond very nearly to the amount produced by the wet assay.

In case the ore contains much arsenic, it will be well to add a few grains of charcoal in the roasting, before the addition of the carbonate of ammonia. In

addition to which it is as well to prepare two crucibles in the same manner, by which means the assay can be verified.

This method I always find to succeed, when moderate caution has been observed, and the assay buttons in both pots will weigh exactly alike.

The wet method of assay I conduct as follows: the ore is to be roasted (100 grs. I find sufficient in cases where the ore is of moderate richness) with the precautions just pointed out, but the operation must be performed in a smooth porcelain, or better still, a platinum crucible. The roasted mass is now to be treated with concentrated muriatic acid, and the whole to be brought to the boiling point; when nearly all the acid has evaporated, water is to be added, and the liquid part, containing the dissolved metal, separated from the mere gangue by filtration. If the roasting has been well performed, one treatment with hydrochloric acid will suffice. The filtered solution is now to have a clean plate of zinc added to it, and heated to the boiling point; in about an hour all the copper will be deposited; the supernatant liquid must be poured off, and the residue well washed with water, dried, and weighed.

I find that the employment of zinc instead of iron in the precipitation, gives a more accurate result: as when the latter metal is employed, the yield is always above that which it ought to be, in consequence of the presence of carbonaceous matter, which is liberated from the iron during its solution, and also particles of metallic iron, which become separated (in consequence of the fibrous texture of the iron employed),

and mix with the precipitated copper. It is true this latter source of inconvenience and inaccuracy, can be removed by treating the mass with dilute sulphuric acid for some time, but of course the more the precipitated metal is washed, the more likely loss is to ensue. The first inconvenience cannot, however, be removed, as the carbonaceous matter cannot be dissolved, so that, on the whole, when accuracy is required, zinc must be employed as the precipitant; and as no more trouble and expense are attached to this method, it is always preferable in use.

It will be seen from the simple nature of this method of assay, that it is much preferable to all those before described, as it takes less time in its execution, there being only very few operations; and it is also much less expensive; and lastly, it is not so injurious to health.

The assay of substances belonging to the Third Class.

The assay of the bodies belonging to this class, is made in precisely the same way as those of the second. They are roasted and fused with a reducing flux. In the ordinary roasting, the greater part of the sulphur and arsenic is given off, but with the aid of charcoal and carbonate of ammonia all may be got rid of; but other metals, as tin, &c., will be reduced with the copper.

In order to be obtained in a state of purity, it must undergo another operation, termed refining: which is described in the treatment of the last class.

The roasting of the ores of this class must be

conducted with much precaution, because they are all so very fusible. There is, indeed, an advantage in obtaining a regulus before roasting, because all the sulphurets are reduced to the minimum of sulphuration, and a very large quantity of arsenic is volatilized, so that the roasting neither occupies so much time, or presents so many difficulties: because, by the loss of sulphur and arsenic, the substance is rendered less fusible.

Assay of substances of this class by the wet way.

The copper contained in bodies of this class can be estimated by the wet way; but the operation is not so simple as with the ores, &c., of the second class. They must be dissolved in aqua regia, diluted with water, and the liquid supersaturated with ammonia filtered, and the precipitate washed with ammonia, until the liquid passing through ceases to be blue. The solution must now be boiled, to expel excess of ammonia, and supersaturated with sulphuric or hydrochloric acids; and the copper precipitated by a plate of iron, or still better, as before explained, by a strip of zinc. If the ore contained silver, it is necessary to filter the solution produced by aqua regia before the addition of the ammonia, because that body would dissolve the chloride of silver formed, which would be reduced by the iron or zinc in the same manner as the copper.

Assay of substances of the Fourth Class.

The substances of this class proceed either from works on the large scale, or from small assays of the

ores of the third class. The proportion of copper in these alloys can be determined either by the wet or dry method. By the wet way, the process is conducted as already pointed out; by the dry way, the operation of refining must be performed.

Refining.—Refining is analogous to cupellation; in fact, it is a true cupellation made on copper; and has for its object the separation of all the metals, with which the copper may be alloyed.

The operation does not, however, give all the copper in a state of purity, and the estimation can only be made approximately. Nevertheless, it is of great use, as it has an analogy with the refining on the large scale, and affords the opportunity of determining the quantity of copper which an alloy would furnish by treatment on the large scale.

The refining of copper is carried on in an ordinary cupel furnace; but as it requires a very high temperature, one possessing more than the ordinary draught must be employed.

The ordinary bone-ash cupel is generally used.

When the furnace and cupels have arrived at the proper temperature, the copper is introduced; when it is fused, lead, if it is judged necessary, is added. The refining then commences. The lead, the alloyed metals, and part of the copper oxidate, and form a fusible combination, which envelopes the circumference of the metallic button, and which the cupel partly absorbs. The button appears agitated, and revolves rapidly, being constantly covered by a brilliant irridescent pellicle. At the instant the operation is about to be finished, the movement becomes quicker, and

the pellicle more shining; then, all at once, the movement and pellicle disappear together, and the button becomes solid.

These phenomena constitute what is termed the *brightening*. When the brightening takes place, the refining is finished, and the cupel may be removed from the fire.

The refined button is covered with a fine crust of protoxide of copper, which cannot be detached without difficulty, if it be cooled slowly; but if it be plunged as hot as possible into water, the oxide is easily removeable by the hammer.

It is generally preferable to sprinkle the button with glass of borax, in the proportion of about 7 per cent. as soon as possible after the brightening; and if the button be plunged into water whilst very hot, the newly formed borate of copper may be detached by the first blow of the hammer. The copper is known to be pure when it is of a fine red colour, and malleable.

In order to arrive at the proportion of copper in the substance assayed, it is not sufficient to weigh the assay button, because a part of it is carried away in the state of oxide, either with the other metallic oxides, or with the borax added to cleanse it.

The alloys of copper submitted to the refining process may or may not contain lead. When they contain none, a tenth of their weight is added successively, until the copper be pure. In order to arrive at the true proportion of copper in the alloy, one eleventh of the weight of all oxidizable metals, (including the lead added), and one tenth of the weight of the

borax, is to be added to the weight of the assay button obtained.*

When the copper alloy contains lead, it may contain just sufficient for the assay, or it may contain too little, or too much. In the former case, there is nothing to add; in the second, lead must be added by tenths, until the copper remains pure; and, in the third, instead of adding lead, a determinate weight of pure copper must be added, the assay concluded as usual, and the quantity added deducted from the quantity obtained, all corrections having been previously made.

The proportion of copper contained in an alloy can be made, however, without making any suppositions as to the quantity of that metal scorified by the lead, by refining as follows: place in two cupels, side by side, in a well heated muffle, 4 parts of pure lead, and as soon as melted, place in one of the cupels, 1 part of pure copper, and in the other, 1 part of the alloy to be assayed, and conduct the refining as usual.

The assay buttons are then weighed; that from the pure copper weighs more than the other; supposing then, that the difference of weight represents the quantity of foreign metals the alloy contained, and that the absolute quantity of copper oxidized is the same in both cupels, it suffices to add to the weight of the button, the loss which the pure copper has undergone, in order to arrive at the proportion of copper contained in the alloy. This supposition is not strictly true, and it seems more probable that

* Berthier.

the exact result is to be arrived at by adding one eleventh the weight of the alloyed metals, and one seventh of the borax to the copper obtained. However, where the alloy is rich, this does not matter very much.

For cupriferous leads, cupel at the same time, 1 part of pure copper, with 4 parts of pure lead on the one hand, and on the other, with 4 parts of the cupriferous lead. The second assay gives more copper than the first, and the difference of weight between the two assay buttons gives the quantity of copper contained in the alloy.

The alloys of copper containing much zinc or tin, cannot be assayed by the process just described, so that they must be treated by the humid method; as, indeed, ought all substances belonging to the third and fourth class.

CHAPTER VIII.

THE ASSAY OF LEAD ORES.

ALL plumbiferous substances which can be assayed by the dry method, may be divided into three classes:—

Class 1, contains all those substances into whose composition enters neither sulphur or arsenic, or mere traces of those bodies.

Class 2, comprises the sulphurets, either antimonial or otherwise.

Class 3, contains those bodies into whose constitution either arsenious, arsenic, or sulphuric acid enters.

As lead is very sensibly volatile, as low a temperature as possible must be employed in its assay. There are some cases, however, that a temperature as high as that employed in an iron assay must be made use of, even at the risk of volatilizing a portion of the lead.

Treatment of ores of the First Class.

The assay of all the substances belonging to this class is very simple, and, in general, offers no difficulty. The operation consists in fusing the substance with a reducing and an alkaline flux, as black flux.

The alkaline flux is useful in allowing the slag to be sufficiently fluid for the formation of all the reduced lead into a single button. Very often the alkaline flux acts on the ganguey matters combined with the ore, and renders them fusible; this happens when silica, or the silicates, are present; but sometimes the action of the alkali is restricted in holding the foreign matters in suspension, forming with them a liquid paste, so liquid, that the globules of lead can fall through it without any difficulty. Lime, magnesia, oxide of iron, phosphate of lime, and finely divided metallic iron, are simply held in suspension in black flux.

To assay nearly pure oxidized substances, as minium or litharge, no flux is absolutely necessary. The operation is then made in a crucible lined with charcoal, and when performed carefully, is very accurate. Nevertheless it is always advisable to add a little carbonate of soda, in order to prevent metallic globules adhering to the sides of the charcoal lining.

As the oxides of lead contain but a comparatively small proportion of oxygen, only a small quantity of reducing flux need be added: 2 parts of ordinary black flux is generally sufficient. There are, however, cases where a larger proportion of flux is necessary, and the quantity of carbonaceous matter in 2 parts of black flux, is not sufficient to effect a total reduction of the metal.

The assay recommended by Berthier is made as follows:

Take 150 grs. of the ore reduced to powder, and mixed well with the proper quantity of black flux,

and the whole placed in a crucible so large, that it is not more than two thirds filled; above this mixture is placed a thin layer of black flux, carbonate of soda, or common salt. The crucible is then placed in the furnace, and gradually heated. As soon as the mixture begins to soften, it boils and swells; and if the heat be not carefully managed, it flows over the sides of the crucible. When the frothing becomes too strong, it must be checked by partially taking the pot out of the fire. After a certain time the boiling up much diminishes; this is in consequence of the reduction being finished, and when no more gas is disengaged, the heat may be raised, in order to finish the assay. The heat to be employed in the reducing process ought not to exceed dull redness; but which may be increased to bright redness, in order to complete the assay. The reason why the heat is raised towards the end of the assay, is to render the flux sufficiently fusible to allow the lead to flow through it.

The crucible is now to be taken out of the furnace, struck lightly on the bottom, in order to collect the lead into a single button. When the slag has been well fused, it presents a concave conical surface. The crucible, when cold, must be broken, the button of lead washed, dried, and weighed. When the assay has been well conducted, the slag contains no globules of lead; it is nevertheless essential to examine it with care. The slag must be pulverized and well washed with water, and the metallic lead separated by decantation. It sometimes happens, that on the sides of the pot, above the fused matter, may be found globules of lead

or yellow patches of litharge, which are produced when the heat has been too great at first. In such a case, the assay must be recommenced.

The method of assay I employ for these substances, does not involve either of these difficulties, as there is little or no boiling up, when the heat is gradually managed, and the slag is always sufficiently liquid to allow all the metallic lead to pass readily through it. I mix well:

200 grs. of ore.
300 grs. of carbonate of soda.
20 to 30 grs. of charcoal.

When the mixture is perfect, the whole is placed in a crucible, and on the top is placed 200 grs. of borax. The pot, in that state, is introduced into the furnace when it is at a very dull red heat, and the heat is gradually increased until it is at bright redness, at which time the flux ought to be perfectly fluid, and the operation terminated. In all cases I make two assays, one to check the other.

The lead obtained in these assays (if the ore contained copper or any other metal) is never pure, if silver, copper, tin, or antimony be present, the whole of either of those substances will be found alloyed with the lead produced; but if the ore contains zinc, and it be heated sufficiently, but a trace remains; nevertheless, the zinc carries off with it some lead.

The following experiments will show what an influence the presence of zinc has upon the return of lead.

100 parts of litharge,
100 parts of oxide of zinc,
300 parts of black flux,

were fused together, and 84 parts of lead were the result.

100 parts of litharge,
100 parts of oxide of zinc,
600 parts of black flux,

were fused together, and but 70 parts of lead were produced instead of 90, which the pure litharge ought to have given. Hence it will be seen, that the more zinc reduced, the more lead is volatilized.

If the oxide of iron be present in the assay, it is reduced, but it remains in suspension in the slag, and the lead does not contain a trace when it has not been too strongly heated. If the assay be made at a very high temperature, the iron may be fused, and then the lead will be ferruginous; this may be ascertained by means of the magnet. A similar result was obtained by many assayers, who thought for a long time that lead and iron could not combine together; but by careful examination, it is easily ascertained that the ferruginous buttons are but mechanical mixtures of lead and iron in grains. Indeed, by careful hammering, nearly all the iron may be removed from the lead, so that it loses its magnetic properties.

The oxides of manganese, when mixed with the ore, are changed into protoxide, which remains in the flux, and is not reduced.

The slags produced in lead assays contain always a

certain portion of that metal. Berthier says that substances containing from 8 to 9 per cent. of lead give 6 per cent. by assay. I have generally found them to give from $7\frac{1}{2}$ to $8\frac{1}{2}$; so that the loss by the process just mentioned is very small indeed. Where, however, very poor substances, such as slags, are to be assayed, the process is best conducted by the wet way.

Assay of lead ores of the Second Class.

The substances containing lead belonging to this class, are either sulphurets or subsulphurets, simple or compound, mixed or unmixed with earthy matters.

Berthier says, that it does not appear to be possible to estimate exactly the amount of lead in a sulphuret by the dry way. It is a fact, that the best methods give a loss of from 6 to 12 per cent. The accuracy of this statement I shall discuss hereafter. Indeed, by some of the old methods of assay, which are yet in use, as much as from 15 to 20 per cent. is lost. This is owing principally to the volatilization of the sulphuret. Berthier, after this, much recommends the employment of the wet process, but I shall show that the dry may be made very nearly as accurate as the wet; indeed, sufficiently so for all practical purposes.

The following is an outline of the processes generally adopted in the assay of lead ores by various persons. The ores to be assayed may be fused :

Firstly, with black flux after roasting.

Secondly, without roasting, with black flux, carbonate of soda, or cream of tartar.

Thirdly, with metallic iron.

Fourthly, with carbonate of soda, or black flux, and iron.

Fifthly, with black flux, oxide of iron, or oxide of zinc.

Sixthly, with black flux, protosulphuret of iron, or sulphuret of zinc.

Seventhly, and lastly, with a mixture of carbonate of soda and nitre.

1*st Process. Roasting and black flux.*—This method is the oldest, and was for a length of time exclusively employed. It is the longest, most troublesome, and least exact of all the known methods.

The sulphuret, reduced to powder, is roasted by heating in a current of air, and continual stirring with an iron rod. The operation is difficult, because the sulphuret and oxide of lead being very fusible, the requisite degree of heat cannot be at all exceeded without the substance softening and agglomerating into small clots, the roasting of which is very tedious and difficult. When the galena is roasted as completely as possible, it is mixed with from two to three times its weight of black flux, and gives only from 66 to 69 per cent. of lead when the galena is pure.

2*nd Process. Fusion with an alkaline flux without roasting.*—In this process the sulphuret is fused with from 3 to 4 parts of carbonate of soda or potash. The crucible it is made in must always be left uncovered. It is slowly and gradually heated until the substance becomes perfectly liquid; the crucible is then to be removed, and allowed to cool; on being broken it will afford, from pure galena, as much as from 75 to even

80 per cent. of lead; one-tenth is always allowed for loss in the assays made in the Hartz.

Instead of an alkaline carbonate, black flux or cream of tartar may be employed in the same proportions. When the latter flux is used, the operation is longer, but the produce is a little greater.

3rd Process. Fusion with metallic iron.—Schlutter and many of the older assayers were aware that iron would desulphurate galena, and ever after advised the addition of a certain quantity of that metal to the different fluxes, which they used in lead assays; but it was at the Practical School of Mines, at Montiers, that iron was first employed alone.

The process is extremely convenient and easy of execution; it always succeeds, and requires no troublesome attention. The fusion takes place quietly, without frothing or bubbling; and as the whole substance employed requires but little space, very small pots may be employed, or a very large quantity assayed. But this process can only be employed for pure galenas, or those which contain at most a few per cent. of gangue.

When galena is heated with iron, the metal is transformed into protosulphuret, from whence it follows, that to desulphurate galena 22.6 per cent. is required; but experience has shown, that it is better to employ a little more, and 30 per cent. can be used without inconvenience. The iron employed ought to be in the state of filings, or wire cut very small. The mixture is placed in a crucible, which is three-fourths filled; the whole is covered with a layer of salt, carbonate of soda, or black flux, and exposed to a full red heat. After the

flux is perfectly fused, the pot may be cooled and broken, and a button is obtained, which at first sight has a homogeneous aspect, but on being struck with the hammer, separates into two distinct parts. The lower part is ductile lead; the upper a very brittle matt, of a deep bronze colour, and slightly magnetic. Pure galena yields, by this process, 72 to 79 per cent. of lead, so that there is a considerable loss, which loss is entirely due to volatilization. Berthier says that it does not appear possible to avoid this loss, which amounts from 6 to 13 per cent., giving as a reason, that it is probable galena begins to sublime before it arrives at a proper heat for decomposition. I shall show this to be ill-founded.

Antimonial galenas, or galenas mixed with iron pyrites, may be assayed in the same manner; but then a sufficiency of iron must be added to reduce the antimony to the metallic state, as well as to reduce the iron pyrites to the minimum of sulphuration. If the galena be mixed with blende, the greater portion remains in the slag, because it is only decomposed by iron at a very high temperature. Blende being infusible by itself, much diminishes the fusibility of the matts produced; and if it exists in very large quantity, it can even hinder their full fusion; in which case, some protosulphuret of iron and metallic iron must be added to the assay, to make the slag more fusible.

All minerals are at a minimum of sulphuration, when existing in matts; from metallurgical works, so much less iron may be used in their assay than if they were pure ores. In very rich lead matts, in which the lead exists as subsulphuret, from 10 to 12 per cent. is suf-

ficient. A small excess of iron may be employed without inconvenience; but if a larger proportion be added than is necessary to execute the desulphuration, the matt contains a large quantity in the metallic state, and loses its liquidity, and in consequence, retains some globules of lead.

4th Process. Fusion with carbonate of soda, or black flux, and metallic iron.—When galena is heated with an alkaline flux, out of contact of air, the slag contains a double sulphuret of lead and the alkaline metal employed; and if iron be thrown into the fused mixture, metallic lead separates, and the iron combines with the sulphur formerly combined with the lead, and the slag will contain a double alkaline sulphuret, containing sulphuret of iron instead of sulphuret of lead. Any earthy substances the ore may contain, will be dissolved by the alkaline flux, without impairing very much its fluidity. All these facts being considered, it may be readily seen that the assay of all earthy bodies, containing sulphuret of lead, may be made in this manner, with as much accuracy as this method of assay can be capable of. Either carbonate of soda or black flux may be employed as the alkaline re-agent, and more of either of those substances must be employed, in proportion to the increased quantity of earthy matters the ore contains. Two parts are nearly always more than sufficient for poor ores, and is best for all cases, because an excess of flux does not diminish the yield of lead; nevertheless, it is sometimes convenient to employ, for the latter class, but half a part. As to the iron, it is employed only to separate that part of the lead which has been dissolved in the state of sulphuret by the alkali, but

not decomposed; so that much less may be employed than is necessary for the decomposition of the whole amount.

Experiment has shown that the maximum amount of lead from pure galena may be obtained by the use of the following mixtures:—

2 parts of black flux, or carbonate of soda, and 10 to 12 per cent. of iron.

1 part of black flux or carbonate of soda, and 20 per cent. of iron.

½ a part of black flux, or carbonate of soda, and from 25 to 30 per cent. of iron.

When black flux is employed, and the iron is in the state of filings, it would be inconvenient to employ too much of the latter, especially if the assay were heated very strongly, because the button of lead might be contaminated with iron; but when carbonate of soda is used with small iron nails instead of filings, the excess of iron is not inconvenient, but rather useful, because the desulphuration is certain to be complete.

The following changes take place in both cases. That portion of iron filings mixed with the carbonate of soda which has not been sulphuretted, is reduced to the state of oxide by the carbonic acid of the alkaline carbonate, and remains combined or suspended in the slag; so that the proportion of iron is never too great, and never becomes mixed with the lead. When black flux is employed, the same oxidation does not take place, on account of the presence of carbonaceous matter, so that the portion of filings not combined with sulphur, and which is merely held in suspension in the flux, passes through it with the globules of lead to the bot-

tom of the crucible; but if instead of filings, small nails are employed, they only suffer corrosion at their surface, without change of form or softening, and after the assay, are found fixed in the surface of the button of lead, so that they can be detached very readily, and without loss of lead.* This, however, I have found no easy task, and have always sustained a notable loss.

There is yet another method of desulphurating lead, by acting on the mixture of assay in iron crucibles; here the mischief of getting a mixture of iron with the lead is avoided; but another error is induced, because the assay, in its heated state, must be poured into an ingot-mould, during which process, some lead is inevitably lost.

5th Process. Fusion with black flux, or carbonate of soda, and the oxide of iron or zinc.—Oxide of iron and oxide of zinc being readily reducible by charcoal, it might be surmised that a mixture of either of these oxides and black flux would be equivalent in effect to metallic iron or zinc, and carbonate of potash, and consequently would answer the same purpose.

When we fuse together—

100 parts of pure galena,
100 to 200 parts of carbonate of soda,
3 parts of iron scales, in powder,
½ part of charcoal,

76 parts of lead were obtained, and a very fluid slag, which retained no metallic globules.

Oxide of zinc produces the same desulphuration, but too much must not be employed, because if much zinc

* Berthier.

be volatilized, much lead will be lost. It is also necessary to add 2 parts of black flux, as the sulphuret of zinc formed is not very fusible.

100 parts of pure galena,
200 parts of black flux,
3 parts of oxide of zinc,

have, in one experiment, 78 of lead and a very fusible flux.

Oxide of iron and oxide of zinc may be employed with carbonate of soda alone, without the mixture of charcoal. It appears that the presence of an alkali determines an action, which would not otherwise take place, between the oxides and galena.

Pure galena affords, with 1 part of carbonate of soda and from 1 to 3 per cent. of iron scales, 73 to 74 of lead. With 1 part of carbonate of soda and 10 per cent. of oxide of zinc, 77 of lead are obtained.

The same result is obtained by means of peroxide of manganese.

6th Process. Fusion with black flux and protosulphuret of iron, or sulphuret of zinc.—Sulphuret of iron and zinc are partially decomposed by the alkaline carbonates, so that a portion of iron and zinc is set free, which forms a compound of an alkaline sulphuret, and sulphuret of iron and zinc. That portion of metal set free is oxidized by the carbonic acid of the alkaline carbonate when there is no charcoal; but remains in the metallic state when a reducing agent is present; but in either case, it re-acts on the sulphuret of lead, and sets lead in the metallic state at liberty. If there be an excess of metallic iron, oxide of iron, or oxide of zinc, they will

remain suspended in the flux, as they are in a state of extreme division, and do not mix with the lead; if there be an excess of metallic zinc, it volatilizes. The following is the result of an experiment conducted on this principle:—

100 parts of pure galena,
100 parts of black flux,
5 parts of artificial protosulphuret of iron,

give very readily 77 to 78 parts of lead. The fusion took place without bubbling or boiling, and the slag was compact, slightly crystalline, and of a metallic black appearance.

The same proportion of lead was obtained by the substitution of blende for protosulphuret of iron; but when only 1 part of black flux is employed, the slag is pasty, and may contain globules of lead. The proportion of flux must be doubled to avoid this.

These experiments show that it is altogether superfluous to employ iron in the assay of matts from metallurgical works which contain much sulphuret of iron or sulphuret of zinc; because by fusion with 1 or 2 parts of black flux, nearly all the lead they contain may be extracted. Iron must be employed, if perfect accuracy be requisite, as I shall hereafter show. Berthier further says: "It shows also, that in order to assay a galena containing much blende, that it need only be fused with 2 parts of black flux."

The same course of procedure does not succeed, however, with galena mixed with much iron pyrites, because the latter forms with alkalies a large quantity of an alkaline sulphuret, which not only holds in combina-

tion all the sulphuret of iron reduced to a minimum, but a considerable portion of sulphuret of lead also; so that but very little metallic lead is obtained; for instance,

100 parts of galena.
100 parts of black flux.
5 parts of natural pyrites

produced but 38 of lead. When pyrites is present, it is necessary to employ metallic iron in the assay. The proportion of which ought to vary with that of the pyrites, and ought to be at least sufficient to reduce the latter to the state of protosulphuret.

7th Process. Fusion with a mixture of carbonate of soda and nitre.—When nitre is made to act upon galena, all its sulphur is transformed into sulphuric acid before the lead is oxidised; and, in consequence, if a suitable proportion be employed, it may be used in the desulphuration of plumbiferous matters. In order to avoid the losses which might be occasioned by deflagration, the ore must be mixed with twice its weight of carbonate of soda, and to extract from the galena the largest possible proportion of lead, from thirty to forty per cent. of nitre must be employed.

This mode of assay is not useful for the estimation of the true quantity of lead, because it gives variable results; and were these results constant it would not be available, because in each assay the quantity of nitre to be employed would have to be guessed.

But it is, on the contrary, an excellent process, and one much to be recommended for the assay of the sulphurets of lead, when the silver they may contain is only to be estimated. The fusion takes place easily,

speedily, and without bubbling. The slag is very fluid, and retains no globules. The proportion of nitre ought to be so managed, that the greatest possible proportion of lead be obtained; but it is especially essential that sufficient be added to destroy all the sulphuret in the slag, which otherwise would retain a notable proportion of silver. There is no inconvenience, however, in the estimation of the silver, by placing an excess of nitre with the mixture; less lead would be obtained; but it would be more pure, and always contain all the silver present in the galena.

Such is an outline of the processes which may be employed in the assay of ordinary lead ores, and containing that metal in a state of sulphuret.

Pure galena contains 85 per cent. of lead, yet it must have been noticed that all the methods of assaying just described, and some of which are in ordinary use, afford in general only 78 per cent. of lead, and in one solitary instance 80 per cent. was obtained. Thus there was a loss of from 5 to 7 per cent. in the best of them. The process which I employ in my laboratory gives as much as 84 constantly with pure galena; and I have obtained, by very careful manipulation as much as 84¾ per cent., thus showing never more than a loss of 1 per cent. and often only that of a ¼ per cent. This process is therefore nearly as accurate as analysis, and is performed in about 20 minutes, and may be conducted as follows:

Prepare two crucibles by smearing their insides (more particularly the bottoms) with black lead, such as used for domestic purposes answers exceedingly well; then place in them, heads downward, two (or three more

commonly) tenpenny nails. Mix 200 grs. of the ore to be assayed very carefully with a like amount of carbonate of soda, and place it in the pot pressing it down tightly about the nails. Then place upon the top of the ore and carbonate of soda, half an ounce of common salt, above that 200 grs. of borax, place the whole in the fire at a red heat for about 10 minutes, and then increase the heat to bright redness for about 10 minutes more, or until the surface of the flux is perfectly smooth. When this happens, take the pot out of the fire, and with a small pair of tongs lay hold of one of the nails and well wash it in the fluid slag until it is perfectly clean and no lead seems to adhere to it, and so on with the two other nails. Then tap the pot gently on the bottom to collect all the lead in one button, and allow it to cool gradually. In taking out the nails, if the operation be performed by raising them with little motion and perpendicularly, very little or no lead, in globules, will adhere to them; but sometimes it is necessary to rinse them well in the flux which is so very fluid that no globules can possibly remain in it, but must sink to the bottom of the pot; it is equally impossible to increase the weight of the button by the accidental presence of a piece of iron, as in the case where iron filings were used; because the nail being much more than sufficient to complete the desulphuration, is rarely corroded at its surface without having any portion of it separated otherwise than as sulphuret of iron which the flux takes up. The button adheres neither to the pot nor the flux; the first on account of the blacklead smeared in the pot, and the latter on account of the peculiar nature of the flux.

The Assay of Galenas containing Antimony.—Sulphuret of lead is often combined with sulphuret of antimony, and the mixture behaves in the manner presently to be described. Either pure lead or lead containing the largest possible quantity of antimony can be extracted at will.

In order to extract pure lead it must be fused with three or four parts of carbonate of soda; then all the antimony remains in the slag partly as sulphuret and partly as oxide; and it is owing to the presence of antimony that the slag will retain no lead. If black flux be substituted for carbonate of soda, the lead obtained contains much antimony, because then that metal cannot remain as oxide in the slag. But in order to separate from the substance assayed, the greatest possible proportion of antimony with the lead, it is necessary to have recourse to the aid of metallic iron. It may be employed either alone or mixed with black flux; in either case the proportion must be exactly determined by repeated guessing. If not enough be added, antimony remains in the slag; if too much, an antimoniuret of iron is formed, which separates tolerably well in matt, but is partly absorbed by the slag.

The following experiments were made by Berthier, on a substance having the following compositions:

Lead	49.8
Antimony	31.0
Sulphur	19.2
	100.0

100 parts of the above compound, which is a double sulphuret of antimony and lead, and

400 of carbonate of soda gave 48 of pure lead.

100 of the double sulphuret.

200 of black flux gave 57 of a semi-ductile lead, which contained at least 7 per cent. of antimony.

100 of the double sulphuret.

200 of black flux.

1 metallic iron gave 60 of a very brittle lead.

100 of the double sulphuret.

33 of metallic iron gave an alloy of lead and antimony weighing 75. The desulphuration was complete in this experiment. The loss is due to volatilization.

Both the lead and antimony can be extracted from an antimonial sulphuret, by roasting and then fusing with 2 parts of black flux; but this operation is unfortunately tedious, owing to the length of the roasting.

Lastly, by fusing an antimonial sulphuret with carbonate of soda and a suitable proportion of nitre, all the lead can be separated in a state of purity; in this case all the antimony is converted into antimonic acid, and the sulphur into sulphuric acid; both of which are found in the slag. When the substance assayed contains silver, that substance alloys wholly with the lead, and none remains in the slag; but when the latter contains sulphuret of antimony, a considerable proportion is retained. On this account this process is preferable to all others.

Assay of Substances of the Third Class.

In the assay of bodies belonging to this class, a reducing agent must be employed; but if that be alone used, the sulphates and arseniates produce sulphurets and arseniurets, and not pure lead. The action of

another re-agent is therefore necessary, in order to deprive the lead of sulphur and arsenic with which it is combined.

There are two re-agents known for the sulphates, and they are the alkaline carbonates or metallic iron; but for the arseniates and arsenites iron must be employed, because the alkaline carbonates have no action on the arseniurets.

In all cases black flux is employed, this furnishes a reducing agent for the oxides, and a flux for the earthy matters. Iron is added when the arsenites or arseniates are assayed; but that metal may either be employed or not, when the sulphates are operated upon. (It is always better to use it).

When a mixture of black flux and iron is employed, the assay is made in exactly the same manner as that of the sulphurets (large nails as recommended in my method of assay are preferable whenever the use of iron is indicated in a lead assay). With the sulphate, the sulphuret of iron formed, combines in the slag with the alkaline sulphuret; but it is not so with the arseniates and arsenites. The arseniuret produced mixes neither with the lead nor the slag; but gives rise to the formation of a brittle matter which adheres slightly to the button of lead.

When only black flux is employed, either of the two following processes may be adopted. First. The ore can be fused with four parts of common black flux; then, as in the case of sulphuret, the excess of carbon determines the formation of a large quantity of an alkaline sulphuret; and consequently produces a desulphuration of the lead. Secondly, It may be fused

with such a proportion of black flux, containing only the requisite proportion of carbon to reduce the oxide of lead, or with an equivalent mixture of carbonate of soda and charcoal. Pure sulphate of lead fused with one part of carbonate of soda and four per cent. of charcoal gives 66 of lead; but in order to employ this method the richness of the ore must be known, and the dry way is then useless, excepting for the estimation of the silver these substances always contain.

Assay by the wet way.—Lead is generally estimated in the state of sulphate of lead in this process. As the sulphuret of lead is the only ore in general use, I shall give its method of analysis by humid re-agents.

The well pulverized galena must be treated by hot weak nitric acid (if strong nitric acid be employed, much sulphate of lead will be formed); when sulphur will separate and a solution of nitrate of lead formed, to which is added a solution of sulphate of soda until no further precipitation takes place. The whole is then to be thrown on a filter, and the residue carefully washed, dried, and weighed; it is sulphate of lead and contains 68.28 per cent. of metallic lead.

If the galena contain either iron pyrites or blende, the analysis is made exactly in the same manner, for solution of sulphate of soda throws down nothing but the lead, leaving the iron and zinc untouched.

CHAPTER IX.

ASSAY OF TIN ORES.

All substances containing tin, which are generally assayed, contain that metal in the state of oxide; such are the ores, either in a rough or washed state, and all varieties of slag.

Although oxide of tin is completely reducible by charcoal at a white heat, it has such an affinity for silica, that whenever that substance is present, the metal cannot be wholly reduced, but at the highest temperature of a wind furnace. Hence the necessity of making all tin assays, at the same temperature as those of iron.

When an ore of tin is heated with a reducing flux, at the temperature of a copper assay, a certain quantity of tin is separated, if the substance is rich; but a large proportion remains in the slag, the quantity of which increases in proportion to the poorness of the mineral; and if a very large quantity of flux were employed, some tin ores which are treated with profit in the large way, would give little or no tin by this method of assay. This was, however, the process employed by most of the older assayers. Schlutter fused at a lead assay heat, with 4 parts of black flux,

and ½ part of resin. Bergman employed 2 parts of tartar, 1 part of black flux, and ½ part of resin.

The following is the result of some of Berthier's experiments on this subject:

Pure oxide of tin gives 72 per cent. of metal, with 4 parts of black flux, in a plain crucible; and 76 per cent. with ½ part of carbonate of soda, in a charcoal lined crucible. Less is obtained in naked crucibles, than in lined crucibles; probably because the alkali of the flux attacks the clay of the pot, forming a silicate, which combines with a certain portion of the oxide of tin employed.

An ore containing 70 per cent. of metallic tin, gave 62 per cent. with ½ a part of carbonate of soda, in a charcoal crucible; 51 per cent. with ½ a part of borax, in a similar pot; 57 per cent. with from 4 to 8 parts of black flux; 60 per cent. with 3 parts of tartar, or ½ a part of carbonate of soda, and one sixth of charcoal, in a plain crucible.

From which experiments may be seen, the advantage of employing an excess of charcoal, and the smallest possible quantity of flux.

The following mixtures of the same ore and quartz, in powder:

Ore .	100	100	100	100	100
Quartz .	25	66	100	150	300

heated with four times their weight of black flux produced. The first, 52 per cent of tin; the second, 43 per cent.; the third, 28 per cent.; the fourth, 10 per cent.; and the last, nothing. The slag contained

from one fourteenth, to one tenth, of oxide of tin, and more in proportion to the quantity of silica.

The slags produced in the treatment of tin ores in the large way, give no return with black flux. Hence this method of assay is defective, and ought never to be employed in the estimation of the quantity of tin.

The above experiments were made at a low temperature: the following results are those obtained by the employment of a high temperature.

Crammer, and after him Klaproth, assayed tin ores at a high temperature; but their processes are long and troublesome.

Before assaying a tin ore, it is usual to submit it to the same kind of operation it undergoes in the large way.

Most assayers roast it with about one fifth of its weight of charcoal, and wash the residue by decantation, until the liquid ceases to be troubled. The object of this operation, is to separate the pyritic substances, which would unite with the tin during its fusion, altering its weight and character; but the same end can be obtained in a more expeditious, simple, and exact manner.

For instance, all pyritic and arsenical substances, being soluble in aqua regia, it is sufficient to boil the pulverized ore with an excess of that liquid, in order to separate all such matters. After the ebullition, dilute with water, throw the residue on a filter, and carefully wash it. The filter is then dried, and the substance removed and calcined, to free it from a little sulphur, which will doubtless be with it, pro-

ceeding from the decomposition of the pyrites. By these means the ore is completely purified, without losing the smallest possible trace of tin.

When the ore has been prepared either by roasting and washing, or by ebullition with aqua regia, it is placed in a charcoal crucible, with the addition of a suitable flux, and assayed at the same temperature as a sample of iron.

The choice of flux depends upon the nature of the gangues. There are two, nevertheless, which may be used in all cases; they are borax and carbonate of soda, employed in the proportion of 30 to 40 per cent., more or less. Borax may be employed in all cases, because it has the property of forming fusible compounds with earthy substances of every kind. As to carbonate of soda, although it has not the same property, it can always be used as a flux for tin ores, because their gangues are very siliceous.

In general, a tin assay may be considered exactly like an iron assay, although the one metal is so much more fusible than the other. Both metals have one property in common, that is, of forming silicates, which are with difficulty reducible by charcoal. In tin assays, in order to be sure no oxides remain in the slag, it is necessary that it contain bases capable of saturating silica. The best flux for tin ores seems to be $\frac{3}{4}$ths of borax, and $\frac{1}{4}$th of lime; and the assay made in a charcoal crucible.

The assay of tin slags must be performed by mixing up a certain weight, as 400 or 500 grs., with from 10 to 15 per cent. of iron filings, and fusing the whole in a naked pot. The iron takes the place of the tin

in the slag, in part, and the remainder alloys with the separated tin, and forms a button which must be analysed by the humid method, which will be presently described.

Analysis by the humid method.

The following method of analysing tin stones, was contrived by Klaproth. A certain amount of the ore in the finest possible state of division, is mixed with six times its weight of caustic potash, and heated to redness in a silver crucible. The fused mass is acted on by means of water and hydrochloric acid; and if any remain undissolved it must be re-fused with more caustic potash, when it will be readily soluble in more hydrochloric acid.

The solution must be evaporated to a pasty consistence, re-acted on by water, and filtered. Silica remains on the filter. A bar of zinc must then be placed in the solution, which will precipitate tin copper and lead. These may be separated by the action of hot nitric acid, which will convert the copper and lead into soluble nitrates, but the tin, into an insoluble oxide, which must be washed, dried, ignited, and weighed; it contains 78.61 per cent. of metallic tin.

The button of iron and tin, obtained by the treatment of tin slag with iron filings, may be analysed by ebullition with concentrated hydrochloric acid. The solution must be filtered from the insoluble residue, if there be any, and treated with sulphuretted hydrogen gas, which will throw down all the tin as sulphuret, without touching the iron. The precipitated sulphuret must be treated with concentrated boiling

nitric acid, which will transform it into peroxide of tin, which must be washed, dried, and ignited. From its weight the per centage of tin is calculated.

Or a bar of zinc may be placed in the solution in hydrochloric acid, which will throw down its contained tin; this process, however, is not so accurate as the former.

CHAPTER X.

ASSAY OF ANTIMONIAL ORES.

ANTIMONIAL substances susceptible of being assayed by the dry way, are divisible into two classes: the first comprises all those bodies containing oxygen or chlorine, and but little or no sulphur; the second, the sulphuret, and all antimonial ores containing much sulphur.

Class 1.—All the oxides of antimony are very readily reduced by charcoal, so that their assay presents no difficulty. The assay is conducted in precisely the same manner as that of oxide of lead, only as antimony is much more volatile than lead, the heat must be managed with much care, and the assay taken from the fire as soon as finished. When all suitable precautions are taken, the loss of antimony is not very considerable; but Berthier says it is never less than from 5 to 6 per cent. This, I think, is too high. Thus the pure protoxide gives 77 per cent. of metal, and antimonious acid 75. The reduction is readily made, without addition, in a charcoal crucible; but when the substance to be assayed is mingled with impurities, some flux must be added. It succeeds equally well with 3 parts of black flux, with 1 part of tartar, with 1 part of carbonate of soda, and 15

per cent. of charcoal, or any other equivalent reducing flux.

When the substance under assay contains oxide of iron, the latter oxide is more or less reduced, and the metallic iron alloys with the antimony.

Oxidized matters which contain but a small quantity of sulphur, can also be assayed in this manner; because the sulphuret gives up to black flux, the small quantity of antimony which it contains, so that but little remains in the slag. The common glass of antimony produces by this method of assay, 70 per cent. of antimony, and occasionally even more than that.

Class 2.—The assay of the members of this class may be made in two ways: first, by roasting and fusing the oxidated matter with black flux; secondly, by fusing the crude ore with iron, or iron scales, with or without the addition of black flux.

The roasting of sulphuret of antimony requires much care, for it is very fusible and volatile, as is also the oxide its decomposition gives rise to. The heat ought to be very low during the operation, and the substance continually stirred. When no more sulphureous acid is given off, we may feel assured that it is perfectly roasted, because no sulphate is ever formed in this operation.

The roasted sulphuret is then fused with 3 parts of black flux, or its equivalent.

Metallic iron separates very readily all the sulphur from sulphuret of antimony; but as sulphuret of iron has a specific gravity very like that of antimony, the separation is very difficult to manage; a strong fire

must be employed when the desulphuration is complete, to keep the whole body in full fusion, for a considerable time. With these precautions two buttons are obtained, which separate very well. The one white, and in large plates, which is antimony; and the other is a bronze yellow, a little brighter than the ordinary sulphuret of iron, because it is mixed with a little metallic antimony. During the operation, a very considerable proportion of antimony is always volatilized, which, by this process, is an inconvenience impossible to avoid.

It is, nevertheless, practised in the large way in some factories, but a good result is not generally obtained. It however appears, that when all the necessary precautions are taken, that it can be employed with profit.

The first precaution which is indispensable is, mixing with the sulphuret only the precise proportion of iron necessary to effect its decomposition, which quantity amounts to about 42 per cent. of its weight. If more be placed, the antimony having a great tendency to play the part of an electro-negative element will combine with the surplus, and an antimoniuret of iron result, part of which will remain in the antimony and part in the slag.

Further, the iron ought not to be rusty, and to be in the finest possible state of division. If the masses be large, a portion of sulphuret of antimony is volatilized before they can be fully attacked. In general 63 per cent. of antimony can be extracted from sulphuret by the aid of iron in the small way, but on the large scale it seems that 55 per cent. is the maximum.

Cast iron cannot be employed instead of wrought, because sulphur has very little action in it. The desulphuration is imperfect, and the slag adheres to the reduced metal.

One of the greatest inconveniences in separating sulphur from antimony, by means of iron, is the strong heat necessary to separate the slag from the metal. This might be remedied by making the slag more fusible and less heavy, by the addition of some flux as an alkaline carbonate or sulphate.

If sulphuret of antimony be fused with an alkaline carbonate and charcoal, regulus is obtained, and a slag composed of an alkaline sulphuret and sulphuret of antimony. If metallic iron be thrown into this slag whilst in fusion, all the antimony separates immediately, and a new slag is formed as fluid as the former, containing sulphuret of iron and sulphuret of the alkaline base employed. If instead of the above process, the iron be mixed intimately with the sulphuret of antimony and carbonated alkali, the result is the same, 100 parts of sulphuret, 42 of metallic iron, 50 of carbonate of soda mixed with one-tenth of its weight of charcoal, or 50 of black flux; give 65 to 66 of regulus with the same proportion of iron, and only 10 of flux, but 62 per cent. can be obtained. In these two cases the fusion takes place very rapidly and without bubbling, and the slag which is very liquid separates readily from the metal. By employing 1 part of alkaline flux the preparation of iron can be reduced from 25 to 30 per cent., and the product of metal is always from 65 to 66 per cent.

Hence in making an assay of sulphuret of antimony it is always better to employ a smaller quantity of iron

than is necessary to complete the desulphuration, and make up for it by increasing the quantity of flux, then it may be insured that no excess of iron will be present.

The alkaline sulphates are transposed into alkaline sulphurets by the agency of charcoal at a slightly elevated temperature. The sulphurets of the alkaline metals by combining with the other metallic sulphurets augment their fusibility very considerably. Thus when sulphate of soda mixed with about one fifth of its weight of charcoal, is added to a mixture of sulphuret of antimony and metallic iron, the metallic antimony separates very rapidly, and the slag almost instantly becomes perfectly fluid.

But it must be noted, that the presence of an alkaline sulphuret diminishes the product of regulus, unless the proportion of iron be augmented at the same time.

For instance with

100 parts of sulphuret of antimony.
42 parts of iron.
100 parts of sulphate of soda.
20 parts of charcoal.

but 22 parts of regulus were furnished; but with

100 parts of sulphuret of antimony.
42 parts of iron.
10 parts of sulphate of soda.
2 parts of charcoal.

62 parts of antimony were easily obtained.

Instead of metallic iron, pure oxide of iron may be used or any ferruginous matter whatever, provided it is rich; but it is necessary to add, at the same time, an alkaline flux and charcoal to reduce the oxide of iron.

Not less than 40 parts of iron scales can be employed

for 100 of sulphuret of antimony, and then on the addition of 50 to 100 parts of carbonate of soda and 8 to 10 of charcoal, about 56 of regulus are obtained; but if with 100 parts of carbonate of soda, from 14 to 15 parts of charcoal be employed, 65 parts of antimony are the result. By augmenting the proportion of scales, that of soda may be diminished. Thus with from 56 to 60 parts of scales, 10 of soda and 10 of charcoal be employed, 58 parts of regulus are the result; and if the proportion of soda be 50 and that of carbon 10, from 65 to 66 and even 67 parts of regulus are obtainable.

The fusion always takes place quickly, and the slags are very fluid.

When sulphuret of antimony is fused with finery slag (silicate of protoxide of iron) carbonate of soda and charcoal, a very white crystalline regulus, in large plates, is obtained; a bronze yellow matter and a black opaque vitreous slag, shining like jet, and in which the greatest portion of the alkali employed appeared to be concentrated. These three substances separate very readily from each other

100 parts of sulphuret of antimony.
80 parts of forge slag.
50 parts of carbonate of soda.
10 parts of charcoal.

Produced very readily 60 parts of regulus.

The best method of assaying sulphuret of antimony seems to be one lately introduced, in which it is mixed with four parts of cyanide of potassium, and heated very gently in a crucible. The heat required in this case is so low, and the operation is made so quickly, that none, if any, of the antimony is lost. So that this process is

decidedly preferable in the way of an assay. In particular methods, however, the wet method must be had recourse to.

The sulphuret of antimony is analysed by boiling with *aqua regia*. The residue consists of sulphur and gangue. It is to be washed and dried; then weighed and ignited. The loss gives the quantity of sulphur, and the remainder is pure gangue.

Water must then be added to the filtered solution, which will precipitate some of its contained antimony as oxichloride which must be separated. The solution must then be saturated with carbonate of potash, and a new precipitate will be formed. The solution is to be filtered, and made slightly acid, then nitrate of baryta must be added to it to separate its sulphur as sulphate of baryta which is to be washed, dried, and weighed; its weight indicates the amount of sulphur, 116 parts are equal to 16 parts of sulphur.

The precipitate by water, of oxichloride which remains on the filter is redissolved by hydrochloric acid, and its antimony separated in the metallic state by means of zinc. The precipitate formed by carbonate of potash can contain lead, copper, iron, and antimony. It must be treated by nitric acid, which dissolves everything but the antimony, which may then be estimated as antimonic acid.

It is always best, before conducting the analysis of sulphuret of antimony, to affuse it with diluted hydrochloric acid, in order to dissolve a portion of the carbonate of lime, which may form part of the gangue. As the composition of sulphuret of antimony is con-

stant, the following process is sufficient in the assay of an antimonial ore.

Boil the ore with concentrated hydrochloric acid, which dissolves only sulphuret of antimony, and precipitate the metal in oxichloride by means of water.

Or after all gangues soluble in very weak hydrochloric acid have been removed, the residue may be weighed, and then acted on by boiling hydrochloric acid, until all action ceases. The residue must be well washed with weak hydrochloric acid, dried, ignited, and weighed, the loss of weight corresponds to the percentage of pure sulphuret of antimony, which contains 72.7 per cent. of metal.

CHAPTER XI.

ASSAY OF CHROMIUM ORES.

The assay of ores of this metal cannot be well made by the dry way on account of its great infusibility, but can be readily accomplished by the wet way. Chromium is always estimated either as oxide or as chromate of baryta or lead.

Its estimation in the state of protoxide is preferable to any other.

The finely pulverized ore must be mixed with from six to eight times its weight of nitre, and deflagrated in a porcelain crucible; when the crucible is cold its contents must be dissolved out by means of hot water, which will contain all the chromium in solution in the state of chromate of potash. The solution so obtained must be filtered, and proto-nitrate of mercury added as long as a precipitate takes place. The precipitate must be separated by means of a filter, and well washed, after which, ignited; all its mercury will be driven off, and pure oxide of chromium will remain, the colour of which is bright green. 100 parts of this oxide correspond to 70 parts of metallic chromium.

ASSAY OF MANGANESE ORES.

The assay of this metal is confined to the amount of peroxide any one of its ores may contain. There are

several methods of effecting this, and the best of these will be described below.

The following method is described in Graham's Elements of Chemistry, page 536.

The value of the oxides of manganese is exactly proportioned to the quantity of chlorine they produce when dissolved in hydrochloric acid, and the chlorine can be estimated by the quantity of protosulphate of iron it peroxidizes. Of pure peroxide of manganese 545.9 parts produce 442.6 parts of chlorine, which peroxidize 3456 parts of crystallized protosulphate of iron. Hence 50 grains of peroxide of manganese, yield chlorine sufficient to peroxidize 317 grains of protosulphate of iron.

Fifty grains of the powdered oxide of manganese to be examined, are weighed out, and also any known quantity, not less than 317 grs., of sulphate of iron. The oxide of manganese is thrown into a flask containing 1½ oz. of strong hydrochloric acid, diluted with ½ oz. of water, and a gentle heat applied. The sulphate of iron is gradually added in small quantities to the acid, so as to absorb the chlorine as it is evolved, and the addition of that salt continued till the liquid, after being heated, gives a blue precipitate with the red prussiate of potash, and has no smell of chlorine, which are indications that the protosulphate of iron is in excess. By weighing what remains of the sulphate of iron, the quantity added is ascertained, say *m* grains. If the whole manganese were peroxide, it would require 317 grs. of sulphate of iron, and that quantity would therefore indicate 100 per cent. of peroxide in the specimen; but if a portion of the manganese only is per-

oxide, it will consume a proportionally small quantity of the sulphate, which quantity will give the proportion of the peroxide, by the proportion as 317 : 100 :: m : per centage required. The per centage of peroxide of manganese is thus obtained by multiplying the number of grains of sulphate of iron peroxidized by 0.317. It also follows, that the per centage of chlorine which the same specimen of manganese would afford, is obtained by multiplying the number of grains of sulphate of iron peroxidized, by 0.2588.

The quantity of oxygen which any peroxide of manganese loses by becoming protoxide, can be arrived at very exactly and in a very convenient manner, by heating it in a finely powdered state, with a solution of oxalic acid. The action commences even in the cold; a part of the oxalic acid is converted into carbonic acid, and an oxalate of the protoxide of manganese is formed. Oxalic acid contains 3 atoms of oxygen to 2 atoms of carbon, since carbonic acid contains 4 atoms of oxygen to 2 atoms of carbon; it may be seen that the oxygen which is estimated, is equal to one-fourth of that contained in the carbonic acid. The carbonic acid is collected as carbonate of baryta, and the operation performed as follows:

Place in a small flask, 1 part of the pulverized mineral, 4 or 5 parts of oxalic acid, and 10 parts of water, adapt immediately to the mattrass a recurved tube, of small diameter, placing its open end into a vessel holding about half a pint of saturated baryta water, which must be frequently agitated in order to favour the combination of the evolved carbonic acid with the baryta in

solution. When the disengagement of gas nearly ceases, the contents of the flask must be made to boil in order to expel all carbonic acid. It sometimes happens that all the peroxide of manganese assayed is not decomposed by the oxalic acid, which can be ascertained if it has not changed colour; in which case, the operation must be repeated.

The following is a method contrived by Dr. Thompson, and is a modification of the one just described. I generally employ it; and when ordinary care is taken, it is at least as accurate as assays made in a more expensive manner, and with more troublesome apparatus.

Take 50 grains of the finely powdered mineral, and place it in a small flat-bottomed flask (capable of standing the heat of a sand-bath), together with about 1½ oz. of water, and a ½ oz. of sulphuric acid. Then place loosely a plug of cotton wool in the neck to absorb any moisture which the carbonic acid evolved in the course of the experiment might carry over. A tube containing dry chloride of calcium may be adapted to the neck of the flask by means of a perforated cork; this method will ensure greater accuracy. The flask (whether fitted up with the tube or cotton wool) containing the water, oxide of manganese, and sulphuric acid, is now to be weighed, and 100 grs. of oxalic acid placed in it, the tube or wool must be replaced, and the effervescence produced be allowed to proceed as long as it will, without the aid of heat; when it ceases, a very gentle heat must be applied for a few minutes, and when cold, the flask must be weighed; the loss of

weight corresponds to the amount of peroxide present. Thus, supposing:

The flask, water, peroxide of manganese, sulphuric acid, and tube or wool, weighed	2000 grs.
Oxalic acid added	100
	2100
And the weight after the operation to be	2060
Loss . . .	40

The sample under assay would contain 40 grs. of peroxide in the 50 grs. of ore employed; hence, the per centage of pure peroxide would be 80.

Fresenius and Will recommend the use of oxalate of potash instead of oxalic acid; but I find the results to be the same whichever I employ; and as the latter is much the cheaper re-agent, it is preferable.

ASSAY OF ARSENIC ORES.

This metal, like the last, cannot be well estimated in the dry way. Its assay is conducted much after the same fashion as that of chromium. A given weight of the finely pulverized mineral must be deflagrated with about ten times its weight of nitre, and the residue boiled with water; the solution will contain arseniate (and if the ore had in its constitution sulphur, which is most likely), and sulphate of potash. Nitrate of lead must be added to the solution (made neutral with nitric acid if requisite) and a mixture of arseniate and sulphate of lead will be thrown down, which must

be well washed and digested with dilute nitric acid, which will dissolve out the arseniate, and leave the sulphate; filter, and saturate the filtered solution with soda, which will throw down the arseniate, which must be washed, dried, and weighed. Every 100 parts correspond to 22.2 parts of metallic arsenic, or 29 parts of arsenious acid, the white arsenic of the shops. This method is only approximative; the following is the better plan to follow.

Digest the ore in strong nitric acid, until nothing more is taken up, and all action on the addition of a fresh quantity of acid entirely ceased, dilute with water, and filter: to the filtered solution add nitrate of lead; collect, and separate sulphate from the arseniate, as before.

The use of nitric acid, in this method of estimation, gives much more accurate results than deflagration with nitre.

ASSAY OF SULPHUR ORES.

This assay may be made in the same manner as an assay of arsenic: either by deflagration with nitre, or by the action of nitric acid; *aqua regia* is still better.

Act upon 100 grs. of the ore by repeated doses of *aqua regia*, assist the action by a gentle heat. When all action has ceased, carefully filter, wash, dry, and weigh the residue; then ignite it; the loss of weight will be sulphur. Add to the filtered solution, chloride of barium, collect the precipitate, wash, dry, and ignite it; every 116 parts of this sulphate of baryta correspond to 16 parts of sulphur. The quantity obtained in this manner, added to that obtained in the first part of

the operation by the ignition of the insoluble residue, will give the per centage of sulphur in the ore.

ON THE ASSAY OF COBALT AND NICKEL ORES.

The assay of this metal, like that of chromium, can only be made by the wet way. The following are some methods of analysis.

The ore must be carefully roasted, adding from time to time a little powdered charcoal; it must then be acted on by *aqua regia* until everything soluble is taken up; the solution must be filtered, and if not acid, made so by the addition of a little hydrochloric acid, and a current of sulphuretted hydrogen passed through it unto perfect saturation; filter, and add to the filtered solution nitric acid, and boil, in order to expel sulphuretted hydrogen; filter, to separate precipitated sulphur. The solution will now contain iron, cobalt, and nickel, which may be separated by the following operations.

Add to the solution an excess of sulphuric acid, evaporate to dryness, and heat to redness; the sulphates of iron and nickel will be decomposed, leaving the oxides of their respective metals, whilst the sulphate of cobalt will withstand decomposition, and can be dissolved out by boiling water; the solution must be filtered, and carbonate of soda added to it, until no further precipitation takes place; the precipitate must be collected on a filter, washed, dried, and ignited; its weight corresponds to the quantity of pure oxide in the ore. To estimate the nickel, treat the insoluble residue with hydrochloric and nitric acids, and add an excess of ammonia; oxide of nickel will be redissolved, and

oxide of iron precipitated. A slight excess of hydrochloric acid is now to be added to the solution of nickel and an excess of carbonate of soda, carbonate of nickel will fall, which is to be washed, dried, and ignited; its weight will correspond to the quantity of oxide of nickel in the ore; 100 parts of this oxide correspond to 78.7 parts of pure metal.

CHAPTER XII.

ASSAY OF ZINC ORES.

ALL bodies containing zinc, which can be assayed by the dry way, may be divided into four classes.

Class 1.—Zinc ores, in which the metal exists as oxide, not combined with silica.

Class 2.—Zinc ores, in which the metal exists, as in the former class, as oxide, but partly or wholly combined with silica.

Class 3.—Zinc ores, in which the metal is partly or wholly combined with sulphur.

Class 4.—Alloys.

Assay of ores of the First Class.

In order to reduce the oxide of zinc contained in substances of this class, it is sufficient to mix them with charcoal, and expose them to a white heat.

At the moment of reduction, the zinc is in a vaporized state. Its vapours, however, are readily condensible, so that the operation may be conducted in an ordinary retort, and all the metal is deposited in the neck without the slightest loss. It seems from this, that nothing is so easy, at first sight, as the assay of an oxide of zinc; but it is not so. It is very easy to reduce all the oxide, but it is not so easy to collect all

the zinc; nor is it easy to condense it all in the metallic state, and in consequence to determine the precise proportion in the ore submitted to assay.

This difficulty consists, firstly, in the deposit being extended over a large surface, and it often adheres very strongly to the sides of the retort, so that it is nearly impossible to detach it; and secondly, as the neck of the retort is open, the air having access to it, brings to the state of oxide all the vapour nearest the end of the neck. The proportion of zinc oxidated is larger in proportion to the smallness of the quantity submitted to assay, and is always very considerable where no more than 200 to 400 grs. are operated upon.

It is not, therefore, in the extraction of the zinc from its oxide that the assay is uncertain, but in its collection.

The distillation of zinc requires a very high temperature, and cannot be performed in retorts of glass; those of earthenware must be employed. It is not necessary to lute these retorts when they are of good quality, and they are better thin, because they heat more rapidly, and are not so likely to crack.

After the mixture of oxide and charcoal has been introduced into the retort, it is placed in the fire. The neck ought to have adapted to it a long tube of glass, with a narrow bore, so as to collect all the zinc which may escape from the wide part of the neck of the retort. This disposition is also convenient, as it does not allow such a free access of air.

It is heated gradually until it is white inside; the zinc is reduced and volatilized, and condensed in the neck; the greater the heat, the nearer the orifice. The

metal can be detached readily from the neck, if it be well black-leaded inside. It is necessary, from time to time, to observe the state of the neck, because when very narrow, it is often obstructed, and if not cleaned out with an iron rod, might cause an explosion.

When the operation is finished, the apparatus is allowed to cool, the retort taken out, placed carefully on its side, and broken; in order, that if any particles of zinc have condensed in its dome, they may be removed.

If the proportion of pure metallic zinc alone be the end, all is collected and fused at a very gentle heat in a crucible, with some black flux. But if the zinc is to be estimated, it must be done in a more exact manner. The deposit must be collected with all possible care, the neck must then be broken to pieces, and every piece having adhering to it either zinc or oxide, must be placed on one side, and digested in hot nitric acid, which takes up those substances. If any be in the glass tube, it must be carefully cleaned by means of acid, and the solution added to that produced by the digestion of the broken neck, and the deposit mechanically collected in nitric acid. The solution is then evaporated gradually to dryness, and heated to redness. The nitrate, by these means, is decomposed, and transformed into oxide, and four-fifths of the weight of which is equal to the quantity of metallic zinc produced in the assay.

The foregone is the method of estimation by distillation, the following is the method of estimation by difference. Two plans of assay in this manner may be adopted: firstly, at an ordinary assay temperature;

secondly, at a very high temperature, as that of an iron assay. In all cases it is necessary to commence with the expulsion of all volatile bodies the ore may contain. If water or carbonic acid alone be present, simple calcination will do; but if carbonaceous matter, roasting must be had recourse to.

When the assay is made at an ordinary assay temperature, it is finely pulverized, and mixed with from 15 to 20 per cent. of equally finely pulverized charcoal, and pressed into a crucible, on which is placed a cover, but not luted, and rapidly heated to whiteness. When no more zinc vapour is disengaged it is cooled, and the mixture in the pot collected. The residue ought to be pulverulent; but as it is mixed with some charcoal, it is roasted, and then weighed. It is evident that the loss represents the oxide of zinc. The charcoal added, it is true, leaves a small quantity of ash, but it is too small to be accounted for.

In making the assay in the manner described, it is to be feared that a small quantity of the oxide remains undecomposed, and that a part of the residue might adhere to the crucible, and could not be detached; and lastly, there is always a state of uncertainty in the state of oxidation; the iron the substance may contain will exist in it after roasting. No inconvenience of this nature presents itself when the assay is made at a very high temperature. This mode is the most exact of all, and leaves nothing to be desired.

The assays of zinc at a high temperature are made exactly as those of iron. They are made in a charcoal crucible, with the addition of fixed fluxes, suitable

to effect the fusion of the gangues mixed with the oxide of zinc, if they be not fusible by themselves. The button is weighed; it is a compound of slag, and grains of iron, which are collected, and their weight ascertained, and by the difference, that of the slag. The weight of oxygen which the iron has lost during its reduction, is then added to it, and by deduction from the substance, the weight of the button and the oxygen so obtained, we have the proportion of oxide of zinc reduced in the assay. On the other hand, by deducting from the weight of the slag the weight of flux added, the weight of earthy substances and irreducible oxides which were mixed with the oxide of zinc is ascertained.

These results can be shown in a tabular form, in the following manner:

Let m be the weight of the crude ore, n the weight of the calcined ore, r the weight of the flux added, f the weight of the cast iron, s the weight of the slag, o the weight of oxygen combined with the iron, calculated from the weight of the metal produced, z the weight of the oxide of zinc, then:

m crude ore = calcined ore . n

r fixed fluxes . . . . . r

$n+r$

Gives metal. . f } Total $f+s$ } $f+s+o$

Gives slag . . s } Oxygen o }

Oxide of zinc $n+r-f-s-o$

Flux added. . r

Earthy matter $s-r$.

The following is an actual experiment by Berthier:

100 crude ore = calcined ore				83.3
10 kaolin acted on by acids				10.0
7 Marble = lime				4.0
				97.3
Gave metal . 45.3	Total . 61.3	Total		80.7
Gave slag . 16.0	Oxygen . 19.4			
		Oxide of zinc		16.6
Fluxes added		14.0		
Earthy matters		2.0		

The above result was confirmed by humid analysis, shewing at once the exactitude of the process.

Assay of ores of the Second Class.

The silicates of zinc are not reducible by charcoal alone, but when in contact with substances which have the property of combining with silica, they are reduced completely, even at a moderate temperature. All the modes of assay just described, for ores of the first class, apply to those of the second, with the exception that the flux, instead of being merely reducing, must have a true fluxing property also: lime or magnesia, are good fluxes.

Assay of ores of the Third Class.

In order to assay the substances containing sulphur, which belong to this class, they must be roasted, and then treated as the ores of the first and second class. Sulphuret of zinc may be roasted without difficulty; and when the operation is made with care, the roasted ore contains neither sulphur nor sulphuric

acid. The only precaution necessary to observe is, that the heat must be carefully regulated at first, in order to avoid fusion which might take place, especially when a certain amount of sulphuret of iron is present. Towards the end the heat may be increased, to decompose any sulphate that may be formed. Both a reducing and fusing substance must be added in this case, as in the last, in order to determine the fusion of the ganguey matters.

Fourth Class—Alloys.

The alloys of zinc, with iron, copper, or tin, may be assayed by heating them in a charcoal crucible, with an earthy flux; and weighing the resulting button, the loss will be nearly equivalent to the quantity of zinc present.

THE HUMID ANALYSIS OF ZINC ORES, ALLOYS, &c.

Calamines may be analysed by placing them in a small porcelain retort, to which a tube, filled with dry chloride of calcium is adapted. The retort is heated to redness, and the tube and retort weighed severally, their respective weights having been ascertained before the experiment. The water is represented by the increase of weight of the chloride of calcium tube. The loss undergone by the retort, represents the quantity of carbonic acid and water.

The residue of the calcination is dissolved in excess of hydrochloric acid evaporated to dryness, and redissolved by water. Some silica remains which has not been dissolved, and which may be collected on

a filter. An excess of ammonia is now formed into the solution, and the precipitate separated by filtration from the solution, which contains all the oxide of zinc. The solution may now be boiled with an excess of carbonate of soda, until all the ammonia has been expelled, the precipitated carbonate of zinc collected on a filter, washed, dried, ignited, and weighed; four fifth of its weight corresponds to metallic zinc.

The analysis of blende may be conducted by treating it with gently warmed strong nitric acid; decant the solution with care, and boil with aqua regia; collect the residue on a filter, and wash. The solution may be treated exactly as in the analysis of calaminiferous ore: with ammonia, and carbonate of soda.

Brass may be analysed by solution in nitro-hydrochloric acid, evaporation to dryness, treating with hydrochloric acid, diluting with water, and precipitating its contained copper by means of a rod of iron, collecting, washing, drying, and weighing the precipitated metal: the loss is zinc.

This is only applicable, however, where the brass contains only copper and zinc; it sometimes, however, contains lead.

CHAPTER XIII.

ASSAY OF MERCURIAL ORES.

The ores of mercury which can be assayed by the dry method, contain that metal, firstly, as metallic mercury, either pure or alloyed; secondly, as oxide; thirdly, as sulphuret, seleniuret, chloride, or iodide. All these substances are assayed exactly in the same way, only some are treated without addition, and others with the addition of a suitable flux.

The ore is distilled in a retort, and the mercury which passes over collected, either in the neck of the retort, or a receiver fitted for that purpose: such as a glass flask kept cool by affusion with water. When but a small quantity is operated upon, say 150 to 200 grs., it is most convenient to use a glass retort, or bent tube retort, heating it gradually over a charcoal fire, taking care to keep the upper part so hot, that no metallic mercury may adhere to it. It must be heated nearly to the melting point of the glass, and until all the mercury has come over.

When the operation is finished, the neck is cut off, weighed, and the mercury detached, and weighed again, the loss of weight is the amount of mercury; or the metal may be detached by means of a feather, and allowed to fall into a basin of water, which, if heated

for a few seconds, will cause the mercury to collect into one globule; the water may be decanted, and the mercury dried, at the ordinary temperature, and weighed.

The mercury wholly condenses in the neck of the retort, under the form of a metallic dew; some may by chance pass off, but in order to prevent such an occurrence, the beak of the retort is plunged into water, or a small dossil of linen, moistened with water, introduced into the neck, the end of which is plunged into water, by which means the neck of the retort is kept constantly cool, and the mercury is found deposited on the linen, from which it may be detached by shaking in water.

When large quantities of substances containing mercury are operated upon, it is necessary to heat very strongly towards the end, in order that the centre of the mass may receive a sufficient amount of heat to effect its decomposition. Naked glass retorts cannot be used; and either coated glass, or porcelain retorts, must be employed. In the large way, as in the distillation of amalgans, &c., cast iron retorts are used.

All substances containing mercury, either in its metallic state or as oxide, are distilled without addition, but with the others it is necessary to employ some re-agent, which will separate and retain the sulphur, selenium, &c.; which re-agent may be a metal, as iron, copper, or tin, or black flux, or a mixture of quick-lime and charcoal: iron filings are most often used. For cinnabar is required about 50 per cent. of iron filings, in order to prevent any of

it being sublimed; the true quantity required is only about 24 per cent., but an excess is necessary, in order, as before stated, to prevent loss: 50 per cent. of iron filings may be employed for the seleniurets, &c. When black flux is used, from about 50 to 70 per cent. is employed. Caustic lime may be employed in the proportion of 30 per cent. mixed with 30 per cent. its weight of charcoal. After the ore to be assayed is carefully mixed with any of the above fluxes, it is always advisable to cover it, when in the retort, with a thin layer of the flux employed, in order to avoid all chance of any loss.

Assay for the amount of Cinnabar in an ore.—The ore to be assayed is distilled, without addition, in a glass retort, and the sublimed cinnabar collected and weighed. The ores containing mercury combined with sulphur, are often mixed with bituminous matters and carbonate of lime; then when an assay is to be made for cinnabar, it often happens that a portion of it is decomposed, either by the carbon present, or by the acid of the bituminous matter and lime, and a little metallic mercury is driven off with the cinnabar. In this case, having weighed the mixture of cinnabar and mercury, the mixture is treated by nitric acid, which dissolves only the latter, and pure cinnabar remains, whose weight is taken, and the quantity of mercury dissolved ascertained by the difference, and from that the quantity of cinnabar calculated, which that quantity of mercury would yield. Every 86 parts of mercury furnish about 100 of cinnabar.

If the gangue of the ore be fixed in the fire, the assay may be made by mere calcination, and the loss of

weight will correspond either to the metallic mercury, oxide, or sulphuret it may contain.

ASSAY OF BISMUTH ORES.

The assay of these ores is made exactly in the same manner as those of lead, only the metal being more volatile, the heat employed must not be so great. Indeed, the chief art in assaying bismuth by the dry way, is to employ the most fusible fluxes, as carbonate of soda, charcoal, and borax, or black flux and borax.

Humid analysis of Bismuth ores.—The finely pulverized ore is treated with nitric acid, until all action ceases; the solution is then filtered, to separate gangue, &c.; add to the solution an excess of sulphuric acid, and boil to dryness; then add water and sulphuric acid, until the solution be perfectly clear, and immerse a plate of clean copper; all the bismuth will be thrown down, when it may be collected, dried, and weighed.

CHAPTER XIV.

ASSAY AND ANALYSIS OF PLATINUM ORES.

PLATINUM and the metals found with it being infusible, as well as the alloys which they form between themselves, they cannot be separated from their stony gangues by the dry way, without combining them with some metal which has the property of forming with them a fusible compound. Tin, copper, lead, and silver are employed for this purpose.

By operating at the temperature of an iron assay at least equal parts of either of the above metals and ore must be employed; but if a lower temperature be more convenient, this proportion of tin, copper, lead, or silver, will not suffice; the ore requires at least twice its weight. The following is the result of some experiments made with the ore of Nischné-Tagilsk.

100 parts of the ore
300 parts of pure copper

400

heated to the temperature of an iron assay, in a charcoal crucible, gave a well fused button, weighing 385 parts. The button was compact, of a deep violet-red colour, susceptible of taking a fine polish, then becoming violet-grey; it was very ductile, hard, and tenacious,

with a fibrous fracture, like copper. The loss being but 25 parts, must be attributed for the greater part to the volatilization of copper, proving that the ore does not contain a notable proportion of oxygen, and that the iron it contains is in the metallic state.

100 parts of the same ore
100 parts of fine silver, in powder

200

covered with a small quantity of carbonate of soda, in a charcoal crucible, gave a well-fused button, weighing 199 parts. It was of a grey colour, intermediate between that of silver and platinum, very ductile and magnetic. The loss here was only 1 part, because silver is less volatile than copper.

According to M. Berzelius, the native osmiuret of iridium does not alloy with either lead, bismuth, or silver, at least at a moderate temperature; but remains in a disseminated state, without having undergone the slightest alteration.

Separation of Platinum and Gold.—Dissolve the alloy in *aqua regia*, add sal ammoniac to the solution, evaporate nearly to dryness at a very low temperature and wash repeatedly with rectified spirit until it ceases to be coloured. The gold alone is dissolved, and when the solution is diluted with water and protosulphate of iron added, metallic gold is precipitated, which must be washed, dried, and weighed; by calcining the ammoniacal chloride of platinum, left by the alcohol, metallic platinum is obtained, which must also be weighed.

An alloy of gold and platinum can also be assayed by fusion with a quantity of silver; the triple alloy

must then be acted on by nitric acid, which dissolves only the silver and platinum, leaving the gold untouched; it must be washed, dried, and weighed.

The platinum is then separated from the silver by the addition of an excess of hydrochloric acid. The silver will be precipitated as chloride, and the solution contain all the platinum, which can be separated from it, as in the last case, by means of sal-ammoniac.

Platinum and Silver may be separated, as just mentioned, when the platinum is in excess; if the silver be in excess, they may be parted by means of sulphuric acid, which will dissolve the silver and not the platinum.

Platinum and Copper may be separated by dissolving the alloy in *aqua regia*, and precipitating metallic platinum by a plate of copper, or by acting on the alloy with nitric or sulphuric acids, which dissolve only the copper.

An alloy of Copper, Gold, Silver, and Platinum may be analysed as follows: The alloy is dissolved in *aqua regia*, at a gentle temperature. The solution is to be diluted with water, and filtered; chloride of silver will remain in the filter, when reduced by boiling with a little zinc and sulphuric acid, the metallic silver may be washed, dried, and weighed.

The filtered liquid must be mixed with its own volume of alcohol, and an excess of sal-ammoniac added; it is filtered, and the deposit being collected, gives the amount of platinum. To the remaining solution is added sulphate of iron and metallic mercury, and the whole well agitated, and warmed in a flask, so that all the precipitated gold may well amalgamate.

The amalgam so produced is washed, dried, and ignited; it gives the amount of gold, the loss is copper.

M. Persoz has found, that by heating platinum ore with a mixture of acid sulphate of potash, and common salt, all its metals are attacked and transformed into chlorides which combine with the chloride of sodium. The action is very energetic, and the mixture must be heated with caution, because the sulphurous acid which is given off might cause the fluid mass to run over the sides of the crucible.

But as the metals which these ores contain are rare and expensive, the smallest quantity ought not to be lost; and even in the extraction of them, the processes of the humid method are made use of. The following is the method proposed by Berzelius.

"The operator first separates mechanically the particles of ore, which differ in appearance. All those which are attractable by the magnet are next removed. Independently of the spangles of metallic iron which were first detected by Osann, the platinum sands often contain metallic compounds of iron and platinum, not only capable of being attracted by the magnet, but possessed even of polarity. These grains have a different composition from those not magnetic, as shown in the two following analyses by Berzelius.

Analysis of the non-magnetic grains.

Platinum		78.94
Iridium		4.97
Rhodium		.86
Palladium		.28
Iron		11.04
Copper		.70
Osmiuret of iridium	in grains	1.00
	in scales	.96
		98.75

Analysis of the magnetic grains.

Platinum	73.58
Iridium	2.35
Rhodium	1.15
Palladium	.30
Iron	12.98
Copper	5.20
Insoluble matters	2.30
	97.86

These grains being separated, their relative proportion is estimated.

The ore is to be treated with diluted hydrochloric acid. The object of this is to free it from the coating of peroxide of iron with which it is often coated, and to dissolve the metallic iron. The quantity of iron separated from the ore in this manner is to be estimated.

The ore must not be ignited until it has previously been weighed; for during the ignition, it generally

acquires a coating of peroxide of iron, and a consequent increase of weight. It is sufficient to dry it upon a hot sand-bath.

The operator must not employ too large a quantity of the ore for analysis. Berzelius thinks about 30 grains is the best quantity. Sometimes, however, when the object is to determine with great accuracy the quantity of a constituent which occurs but in a very small relative proportion, a larger quantity of the ore must be dissolved. But in such a case, every other constituent is to be neglected.

Berzelius determines the solution of the weighed metal by *aqua regia*, in a glass retort furnished with a receiver, which is kept constantly cool. The acid distilled over during the solution is yellow, which colour does not proceed merely from the presence of chlorine, but from the constituents of the solution which are carried over mechanically.

The acid is distilled until the liquid has a syrupy consistence, and congeals on cooling. The saline mass so formed is dissolved in the smallest possible quantity of water, and the solution is poured off with all due precaution. The acid distilled over into the receiver is poured upon the undissolved portion of the ore in the retort, and again distilled. The second distillation generally effects the complete solution of the platiniferous matter.

If the distilled liquid be not colourless it must be returned into the retort and redistilled. The residue must be evaporated to a syrupy consistence as before, and treated with water. The distilled liquid generally

contains a small portion of peroxide of osmium, of which a part is lost by the re-distillation; its quantity, however, is in general very small.

The colourless distilled liquid is diluted with water, and saturated either with ammonia or with hydrate of lime. The acid, however, must remain a little in excess. The object of this saturation is to prevent the decomposition of the sulphuretted hydrogen gas, with which the solution is afterwards to be precipitated.

The precipitation is to be made in a flask which can be closely stopped, and of such a size as to be nearly filled with the solution. When the solution contains free hydrosulphuric acid, the flask is stopped and left to itself until it is perfectly bright, which will be in about two days. The clear liquid is removed by a pipette, and the sulphuret of osmium collected in a weighed filter in which it is washed, dried, and weighed. According to theory the resulting sulphuret of osmium should contain 60.6 per cent. of that metal; but it is not obtained free either from moisture or excess of sulphur, it is also slightly oxidized during the process of drying. According to some experiments made by Berzelius with weighed quantities of this substance, it appears that the sulphuret of osmium obtained by the operation just described, contains from 50 to 52 per cent. of osmium. In general, however, the quantity of osmium is so small that an error of a few hundredths in the reckoning, of the quantity of osmium contained in this preparation is of no importance in regard to the analysis.

Respecting the metallic solution from the retort, it

sometimes happens that after the saline mass has been dissolved in water, it smells slightly of chlorine.

This happens through the decomposition of a portion of the chloride of palladium. The solution must be allowed to digest until it no longer smells of chlorine. If the solution became troubled during the digestion, a portion of oxide of palladium is precipitated, which must be redissolved. The solution is filtered through a weighed filter, upon which is collected that portion which is undissolved. This portion consists of grains of osmiuret of iridium, of spangles, of the same alloy, and of grains of sand, which could not be separated mechanically before the analysis. Sometimes, in addition to these, a black powder is found, which has the appearance of charcoal, and capable of passing through the filter during the washing of the other grains. This is peroxide of iridium, and is due to the presence of too much nitric acid in the *aqua regia*. As this occasions much extra work in the analysis, an excess of hydrochloric acid must be employed in making the *aqua regia*.

The filtered solution is now mixed with twice its bulk of alcohol, specific gravity .833; to that the mixture may contain about 60 per cent. of its volume of alcohol. A very concentrated solution of chloride of potassium is now added, as long as it determines any precipitate.

The precipitate consists of the double chlorides of potassium and platinum, and of potassium and iridium, contaminated with that of rhodium, and a little of that of palladium.

The precipitate has a fine lemon yellow colour when

it is free from iridium; but when iridium is present it presents all shades, from deep yellow to cinnabar red.

It is placed upon a filter and washed with a mixture of alcohol (containing about 60 per cent. of anhydrous alcohol) and a small proportion of concentrated solution of chloride of potassium. The precipitate must be washed until the liquid passing through gives no precipitate with sulphuretted hydrogen.

The analysis is now divided into two distinct parts. The examination of the washed precipitate A, and treatment of the alcoholic liquid B.

A. The washed salt is dried and carefully mixed with an equal weight of carbonate of soda. The filter with that portion of the precipitate which it is impossible to separate from it must be burnt, and the ashes mixed with a little carbonate of soda, and added to that mixed before. The whole is very gently heated in a porcelain crucible, until the mass is black through and through.

By acting thus the double salts are decomposed, and the platinum, whose oxygen passes away with the carbonic acid, is reduced. The rhodium and iridium meanwhile become oxidated, and remain in such a state as to permit of their separation from the platinum by solution.

When instead of following the process just recommended, the precipitation of the double salts is effected by muriate of ammonia, the heating of the precipitate in a crucible not only reduces the platinum but the rhodium and iridium also, so that on treating the heated mass with *aqua regia* all three are dissolved.

The heated saline mass is washed with water until the greater mass of the saline contents is dissolved;

diluted hydrochloric acid is then added to the remainder to extract the alkali combined with the oxides of iridium and rhodium. The mass is washed, dried, and ignited. The filter may be burnt and an allowance made for the weight of the ashes; but it is to be noted that the filter must be burnt by itself, lest the metallic oxides be reduced. The mass is afterwards weighed.

When this is done the mass is mixed with five or six times its weight of bisulphate of potash, and fused in a platinum crucible. During the ignition the rhodium dissolves, and its solution is accompanied by an evolution of sulphurous acid gas. The platinum crucible must be kept closed during the ignition, by a cover which fits well, to check the too rapid volatilization of the acid. As soon as the saline mass becomes fixed and crystalline at the surface when the cover is removed, the crucible must be taken from the fire and cooled. The salt is then dissolved in boiling water, and the undissolved residue is treated again with bisulphate of potash. The melted salt is red and transparent when it contains but little rhodium, but appears dark and black when it is nearly saturated with the metal. So long as the salt continues to receive colour the re-melting must be repeated.

In order to avoid in analysis the employment of too large a quantity of bisulphate of potash, the operator may supply sulphuric acid as follows. When the bisulphate of potash appears to have lost the greater part of its free acid, weighed portions of the distilled sulphuric acid may be added to the mixture. The whole cautiously heated until the water of the acid be expelled, and the fusion thereupon be continued.

The quantity of rhodium can be determined by two methods. According to the first, the undissolved platinum is washed, ignited, and weighed, and the quantity dissolved is equal to the peroxide of rhodium which contains 71 per cent. of metal; or the washings which contain the rhodium are super-saturated with carbonate of soda, evaporated to dryness, and ignited in a platinum capsule. If the mass be now acted on by water, peroxide of rhodium will remain. It may be collected in a filter, washed, dried, ignited, and finally reduced, by means of hydrogen gas. The resulting metal is weighed. The rhodium thus obtained, sometimes contains palladium. This is extracted by *aqua regia.* The solution of palladium is then neutralized and precipitated by cyanide of mercury, which may be washed, dried, and ignited. The residual mass is metallic palladium, which may be weighed.

After the separation of the rhodium, the metallic mass is treated with very weak *aqua regia,* by digestion with which pure platinum is dissolved. The solution has a very deep colour which is owing to the peroxide of iridium in suspension; but when it has become bright by deposition it has a pure yellow colour. It is then decanted, and concentrated *aqua regia* mixed with chloride of sodium poured upon the residue. The solution is now evaporated to dryness. The addition of the chloride of sodium is to hinder the production of protochloride of platinum. A small quantity of iridium is dissolved in the very concentrated acid; but, if it were not used, a considerable portion of platinum would remain mixed with the iridium.

When the dry mass is acted on by water, peroxide

of iridium remains unacted upon. If it were washed with pure water to dissolve out all the platinum, it would be carried through the pores of the filter, to prevent which a dilute solution of chloride of sodium must be employed, and to remove the least traces of that, solution of muriate of ammonia is used. The filter is now to be burned, and the peroxide of iridium remaining, with its ashes, reduced to the metallic state by a current of hydrogen gas, and weighed.

The solution of chloride of sodium containing a small quantity of iridium is mixed with carbonate of soda, dried, and ignited. The product freed from soda salts by water, and from platinum by weak *aqua regia*, leaves peroxide of iridium, which must be reduced to the metallic state, and added to that already obtained.

In order to arrive at the weight of platinum, the operator must deduct the weight of the peroxide of rhodium, from the united weight of the peroxide of rhodium, peroxide of iridium, and platinum. He must then add to the weight of the iridium obtained, 12 per cent. of the weight of that metal, to produce the weight of peroxide of iridium which must be deducted from the weight of the platinum.

The reduction of the platinum from its solution, would only increase the length of the operation, without adding anything to its accuracy.

B. Treatment of the alcoholic solution. This solution is poured into a flask capable of being well stopped, and sulphuretted hydrogen passed in unto saturation. It is then stopped, and allowed to remain at rest for twelve hours in a warm place; at the end of which time all its metallic sulphurets will be precipitated.

Sometimes the solution is red, owing either to the presence of rhodium or sesquichloride of iridium.

The solution must now be filtered and evaporated to expel all alcohol, during which operation a little more metallic sulphuret will be precipitated, and which must be added to that already obtained. The mixture of sulphurets thus obtained, consists of the sulphurets of iridium, rhodium, palladium, and copper; while the filtered solution contains iron, rhodium, iridium and a trace of manganese. During the evaporation of the alcohol, a greasy-like metallic sulphuret of a disagreeable odour is deposited, which cannot be washed out. After the solution has been entirely washed away from this substance, it can be dissolved by pouring a little caustic ammonia into the capsule. The solution is now poured into a platinum crucible, and evaporated to dryness. The moist metallic sulphurets are then placed in also, and roasted until all sulphurous acid is expelled. On the cessation of roasting, concentrated hydrochloric acid is poured over the mass; which, owing to the solution of subsulphate of copper and palladium is coloured green or yellowish green. Oxide of iridium and rhodium with a little platinum remain unacted upon.

The solution in hydrochloric acid is mixed with chloride of potassium and nitric acid, and evaporated nearly to dryness; a dark saline mass is the result, and which is composed of chloride of potassium and cupreo-chloride of potassium, with palladio-chloride of potassium. The two first of these salts are dissolved out in alcohol, specific gravity .833, and the palladium salt is placed on a filter washed with alcohol of the

same specific gravity. It contains 28.84 per cent. of palladium when dried and ignited.

The spiritous solution, which contains the copper salt, is evaporated to get rid of alcohol; and the contained copper is precipitated, either by means of pure potash or by adding sulphuric acid and a plate of zinc.

That portion of the roasted sulphurets which was insoluble in hydrochloric acid, is fused with bisulphate of potash until it ceases to become coloured. The mixture, in this case, contains much more rhodium than the precipitate obtained at the commencement of the analysis. The residue undissolved by bisulphate of potash, which is peroxide of iridium with a little platinum, is treated with *aqua regia*, and the peroxide reduced by hydrogen gas as stated in a former part of the analysis.

The concentrated solution from which the sulphurets were precipitated, contains only iron in a state of protochloride with a small quantity of iridium and rhodium with a trace of manganese. It must be mixed with a proper quantity of nitric acid, and boiled till the iron be fully oxidized. The peroxide of iron is then precipitated by caustic ammonia, and the precipitate washed, ignited, and weighed.

This peroxide of iron, however, contains a small quantity of iridium and rhodium, to separate which, after weighing the peroxide, it must be reduced by hydrogen gas. The reduced metal is treated with hydrochloric acid to dissolve iron, and the black undissolved portion is collected on a filter, ignited with exposure to air and weighed, its weight deducted from that of the peroxide of iron previously obtained, leaves

the quantity of the latter in a pure state. The solution filtered from the precipitate by ammonia, is mixed with carbonate of soda in sufficient quantity to decompose the ammoniacal salts, and evaporated to dryness. On treating the residue with water, after a gentle ignition, peroxides of iridium and rhodium remain undissolved, but they are generally too small for separation.

CHAPTER XV.

ASSAY OF SILVER ORES.

All argentiferous substances may be divided into two classes:

Class 1.—Bodies in which silver is mixed, or combined with substances, not capable of being cupelled: such, for the most part, are sulphurets, seleniurets, arseniurets, alloys with antimony, tin, &c.; slags, cupels, bottoms, dross, litharge, and ferruginous, and earthy ores.

Class 2.—Substances capable of direct cupellation: as alloys of lead, copper, or copper and platinum; the sulphuret and seleniuret, galena, sulphuret of copper, with some arsenio-sulphurets, and the chlorides.

The following are principally Berthier's processes in this assay, as in that of gold also. These I always employ myself, and am convinced that they cannot be surpassed in accuracy by any other known method of operating.

In all assays of silver, it is necessary to obtain an alloy of that metal with lead, and then submit that alloy to cupellation in order to separate the lead, and all other foreign substances with which the silver might have been contaminated.

Class 1.—In order to separate silver from this class of substances, a plumbiferous alloy must be formed. The different methods by which this object can be attained, are the following: firstly, fusion with a reducing flux; secondly, fusion with oxidating re-agents; thirdly, scorification; fourthly, amalgamation; fifthly, fusion with a reducing flux.

All substances containing lead in the state of oxide, such as carbonates, phosphates, &c., are fused directly with a reducing flux, as also are slags, old cupels, litharge, &c. All plumbiferous sulphurets, &c., containing silver, are assayed as for lead by the processes already pointed out, taking care to follow the method which gives the largest proportion of lead.

All argentiferous minerals containing copper may be assayed as copper ores; because an alloy of copper and silver can be cupelled by means of lead.

In making assays of silver with lead or copper, it is sometimes necessary to commence the operation by roasting the ore; under other circumstances, also, argentiferous matters are roasted.

There is nothing very particular to be observed in this roasting, the temperature alone requires attention in managing the temperature well at the commencement of the operation, in order to avoid softening, and especially to avoid a very rapid disengagement of arsenical vapours, because a very considerable amount of silver may be lost by that means.

All substances which contain reducible oxides are fused with a reducing flux, as also those from which charcoal separates metals only which alloy with lead, or metals which do not hinder the process of cupellation;

but it is necessary to add to the reducing flux a certain proportion of litharge, in order to produce metallic lead, with which the silver may alloy. A mixture of metallic lead and any flux may be substituted for that of litharge and a reducing flux, but the latter is preferable, because the lead produced is uniformly diffused throughout the whole mass of flux, &c., not allowing a particle of silver to escape its action.

The reducing agent employed in nearly all assays is charcoal, either in its ordinary state, or as it is found in black flux. Starch and other analogous substances may be, as before mentioned, substituted for it. The proportion employed must be varied according to circumstances, so that the silver-lead produced be not too rich, or that too great a proportion of lead be reduced. If the silver-lead be too rich, much of the precious metal may be lost in the slag, and if too great a quantity of lead be produced, silver is again lost, owing to the long exposure to the fire during cupellation; and indeed this is the most fruitful cause of loss, for more is lost in this manner than by having too little lead produced. In order to know the right proportions, the following data will serve as a guide: 1 part of charcoal produces about 30 parts of lead with litharge, and 1 part of black flux produces about 1 part of lead.

The fluxes employed in this kind of assay are litharge, black flux, carbonate of potash or soda, and borax. Litharge is an exceedingly convenient flux, because it occupies very little room, and fuses without bubbling, producing very liquid scoriæ with nearly every substance. Experiment has shown that nearly all argillaceous, stony, and ferruginous substances fuse very

well with from 8 to 12 or more parts of litharge. If from ½ to 1 part of black flux, or ⅒th to ⅛th of charcoal, be added to 1 of ore, from ½ a part to 1 part of silver-lead will be produced.

Black flux is employed in the fusion of all substances containing a large proportion of alumina, or in which lime is the predominant substance; from 2 to 3 parts of this flux generally suffice; 1 part of litharge is added to the assay, which is wholly reduced, producing nothing but lead.

The carbonates of potash or soda produce exactly the same effects as the alkali of the black flux. A certain quantity of charcoal must, in this case, be added to the assay.

Schlutter fuses the poor refuse of goldsmith's workshops, mixtures of fragments of crucibles, glass, &c., with 2 parts of carbonate of potash, when they are very earthy, and with 1 part only when they contain much glass, adding, at the same time, to the mixture, a little litharge and granulated lead.

Borax has, like litharge, the advantage of being an universal flux; it is useful especially for the fusion of substances containing much lime, such as the scrapings of old picture frames, &c.; but it is necessary to take great care in the assay, in order to avoid the loss which its boiling up might occasion. This only applies, however, to its use in its ordinary state; if previously fused, that is, used as glass of borax, no particular care need be taken.

FUSION WITH OXIDATING RE-AGENTS.

Litharge.—The oxidating agents employed in the assay of argentiferous substances, are litharge and nitre. Litharge attacks all the sulphurets, arsenio-sulphurets, &c., and oxidizes nearly all the elements, excepting silver, when employed in sufficient quantity, and a quantity of lead equivalent to the oxidizable matters present is reduced, so that there results from the assay a slag containing an excess of oxide of lead, and an alloy of lead and silver, very little contaminated with foreign metals, if no copper be present, and which can be submitted directly to cupellation. This method of assay is exceedingly convenient and quick.

The pulverized mineral is well mixed with litharge, and the mixture placed in a crucible, which may be very nearly filled, as there is scarcely any boiling up when the pot and its contents are submitted to the fire. A thin layer of pure litharge is placed above the mixture, the whole is then heated rapidly, and as soon as the litharge, &c., are completely fused, the crucible is taken from the fire. It is inconvenient to heat it for any length of time, on account of the corrosive action litharge has on the substance of the crucible, which it rapidly destroys.

The preparation of litharge which must be employed, depends upon the nature and quantity of oxidizable matters present in the ore. It ought to be in general very great, because, so that the slag may not contain the least trace of silver, it is absolutely necessary that no sulphurous matters be present. But it is known how much litharge is required to decompose the metallic

sulphurets. Pyrites require about 50 parts, mispickel, blende, sulphuret of antimony, copper pyrites, grey cobalt, and copper, require from about twenty-five to forty times their weight. For sulphuret of bismuth, 10 is sufficient, and for galena or sulphuret of silver, but 4 or 5 parts need be employed. The proportion of litharge cannot be so great for a mineral containing much stony gangue as for one entirely metallic. Experiment has proved, that in the assay of rough schlichs, such as those treated in the large way by amalgamation, can be made very exactly with from 10 to 12 parts of litharge.

Alloys of silver with the very oxidizable metals, can be assayed by means of litharge, such as those of iron, antimony, tin, zinc, &c.; but in order to have a successful result, the alloys should be reduced to a very fine state of division, so that they must be at least granulated; and it is very often necessary to repeat the operation several times on the fresh alloy of lead produced.

The method of assay just pointed out is inconvenient, on account of the large quantity of lead it produces; pyrites giving 8½ parts, copper pyrites and blende, 7 parts, sulphuret of antimony and grey copper, about 6 parts, &c. In order to avoid this inconvenience, part of the oxidation can be performed by means of nitre. Nitre alone, employed in excess, oxidizes all metallic and combustible substances found with silver, and even under certain circumstances, a portion of silver itself; but when the proportion is insufficient to oxidize the whole, and when the mixture contains at the same time litharge, after the nitre has produced its action, the

litharge acts in its turn on the remainder of the oxidizable substances, and the resulting lead carries down the silver set free. So that by employing suitable proportions of nitre and litharge, all the silver contained in oxidizable minerals may be extracted, and any quantity of lead required may be thus alloyed with it.

As to the requisite proportion of nitre, it can be come at by practice, aided by the following data. It requires about 2½ parts of nitre to completely oxidize iron pyrites, 1½ for sulphuret of antimony, and ⅔ for galena.

This determination can be ascertained at once as follows: Fuse 1 part of the mineral with 30 of litharge, and weigh the resulting button of lead; and having fixed upon the quantity of lead necessary to carry on the cupellation properly, deduct it from the whole weight of the button, and the difference will be the amount of lead necessary to leave in the slag in the state of oxide; and as it has been proved by experiment, that 1 part of lead requires .25 to .30 of nitre; that is, from 25 to 30 per cent., it is easy to calculate the quantity necessary to be added.

When the ore contains sulphur, the latter forms with the nitre sulphate of potash, which swims on the slag without combining with it.

The assay of silver ores, by means of nitre, is advantageous and useful in a variety of cases. If we wish to determine, for example, very exactly the per centage of silver in a poor galena, a large quantity, say ¼ of a pound must be fused with about an ounce or an ounce and a half of nitre, and a quarter of a pound of carbonate of soda, or better still the same quantity

of litharge, one of either of which must be employed to flux the gangue and temper the deflagration. After the fusion, all the contained silver will be found alloyed with a very small quantity of lead.

Sometimes the assay is made with a larger quantity of nitre than is requisite for the oxidation, and when the mixture is perfectly fused, a certain quantity of metallic lead is added, taking care to cover the whole surface of the mixture, either by using granulated lead or a convenient mixture of litharge and charcoal, or litharge and galena. The shower of metallic lead passing through the fluid mass, alloys with all the silver it finds in its passage, and so concentrates it.

This process, however, cannot always be confidently employed. If an excess of nitre be employed with substances susceptible of forming peroxides capable of attacking silver, such as some cupreous substances, the lead added reduces the greater part, but not the whole of the silver in the ore, so that the assay will not be perfect.

Scorification. — Scorification has, like fusion with litharge, the effect of producing an alloy of lead capable of cupellation, and a very fusible slag composed of oxide of lead, and all the matters foreign to silver, converted into the state of oxide. In the fusion the oxidation of these substances takes place by the action of the litharge, which furnishes at the same time by its reduction the lead necessary to form the alloy, whilst in scorification all the substances susceptible are oxidized in the roasting by means of the oxygen of the air, and the litharge itself is produced by the oxidation of part of the lead mixed with the ore to be assayed.

In this operation vessels termed scorifiers are employed; they are made of very refractory earth, and are in shape somewhat like a deep saucer. They are heated in the muffle of the cupelling furnace, and as many assays may be made at one time as the muffle holds scorifiers.

Before introducing the scorifiers into the muffle, a given weight of the ore reduced to powder is mixed intimately with a certain quantity of granulated lead and placed in each. They must then be heated gradually for about a quarter of an hour, with the door of the muffle closed, in order to fuse the lead, then diminish the heat and allow access of air by opening the door. The current thus established in the muffle soon causes the roasting to commence, and this roasting goes on without its being necessary to continually agitate the mass, as in the case of pulverulent substances.

During the oxidation, a slag is formed in the fluid metal, which is thrown towards the edges, and which by continually augmenting, at last entirely covers the bath. This slag, which is often solid at the commencement, becomes softer and softer, and at last becomes perfectly fluid; because, in proportion to the advance of the operation, the proportion of oxide of lead continually increases. When it is judged that the scorification has been carried far enough, the melted matter is stirred with a rod of iron, in order to mix with the mass the hard or pasty parts attached to the bottom or sides of the basin. The fire is then urged so as to completely liquefy the slags. It may be ascertained when they are sufficiently fluid, by plunging into them a red hot iron rod, which must only be covered with

a slight coating, capable of running off, and not solidifying into a drop at the end.

This condition of liquidity is indispensable, in order to enable the metallic globules to unite into a single button. When this end is not attained, it is because the scorification has not been carried sufficiently far; or, because a sufficient quantity of lead has not been added to form the flux, in which case a fresh quantity must be added, or what is preferable, the assay recommenced with larger proportions.

When the operation is finished, the scorifier must be removed, and its contents immediately poured into a circular or hemispherical ingot mould. The metallic particles fall to the bottom, and as the cooling proceeds, they form a button covered by the slag, which is readily detachable by a blow of a hammer; it ought to be very homogeneous and vitreous, and its colour varying from brown to greenish.

It is always advisable to examine it, and ascertain if it contain metallic globules. The button ought to be as ductile as ordinary lead, if not it cannot be cupelled, and must be submitted to a fresh operation. It is in general advantageous to push the scorification to its greatest extent, because experiment has proved that less silver is lost than when a large button is cupelled. Nevertheless, there is a limit, because if the silver-lead produced be too rich, the least loss in the shape of globules would cause a notable one in the silver. Besides, as litharge exercises a very corrosive action on earthy matters, if the scorification be continued for a great length of time, it sometimes happens the vessel is pierced, and the assay has to

be recommenced. The button of lead remaining ought to weigh about 200 grs., when the ores treated are of ordinary richness. The length of time a scorification takes is, from half an hour to one hour. The scorifier can be rendered less permeable to the litharge, by being rubbed inside with chalk or blood.

There may be distinguished three distinct periods in the operation, viz: the roasting, the fusion, and the scorification. At first a strong fire is employed; but the doors of the furnace are opened, as soon as the mixture is fused. The mineral, being specifically lighter than the lead, is then seen floating on its surface, or forming masses in it; the roasting then commences, and from the appearance of the vapours, the nature of the combustible matter it contains may be judged. Sulphur produces clear grey vapours; zinc, blackish white vapours, and a brilliant white flame; arsenic, whitish grey vapours; antimony, fine red vapours, &c. When no more fumes are seen, the mineral has disappeared, and the fused lead perfectly uncovered, the roasting has terminated; this generally requires from eighteen to twenty minutes. At this time the fire is urged, so as to cause all the substances in the scorifier to fuse. It can be ascertained that the fusion is complete, by the following signs: at the instant the muffle is opened, the button becomes whitish red, with a greyish black band, and there arises from the melted mass, clear white fumes of lead, and the slag appears like a ring encircling the metal. The third period then commences: the furnace is cooled, as in the roasting, and the lead is allowed to scorify, until it is entirely covered with fused oxide;

this last period generally lasts about fifteen minutes. The fire is then increased for about five minutes, and the contents of the scorifier poured into the mould.

The process of scorification is applicable to all argentiferous matters, and is at the same time the most exact method of assay, as also the most convenient, when a large number of assays are required at the same time, because they are entirely executed in the muffle, which, with most assayers, is generally hot.

When the silver ores are stony, the oxide of lead, formed during the roasting, combines with the gangue, forming a fusible compound, whilst the remaining lead alloys with the silver. When the ores are metallic, the oxidizable bodies absorb oxygen from the atmosphere, and the oxides so formed, combine with the litharge produced at the same time, forming a compound which becomes very fusible, in proportion as the oxide of lead increases up to a certain time, and if the scorification has not been pushed sufficiently far, the button will contain, besides silver and lead, a little copper, which will not, however, interfere with the cupellation. There is this one peculiarity about scorification, that however small the proportion of lead may be that is used, at the end of the operation the slag does not contain any oxysulphuret. For instance, even when oxysulphurets are produced in the course of scorification, they are completely decomposed in the roasting, and in consequence it is very rare that the slag retains any proportion of silver; and as to the proportion of lead employed, only just

enough to render the slag liquid, and to produce sufficient lead for cupellation, is necessary.

It is different, however, when the sulphurets and arsenio-sulphurets are assayed by means of litharge, for from 30 to 50 parts of that substance must be employed to prevent the scoriæ retaining any silver.

All scorifications may be conducted by the simple addition of lead; but it has been proved that the operation proceeds more quickly, and with less danger to the scorifier, when borax is employed. This salt dissolves the oxides in proportion as they are produced, as also the gangues, and forms a very liquid slag from the commencement of the operation, which does not happen when lead alone is used, because litharge, which can alone cause the fusion, is not present in the slag in sufficient proportion, but at a very advanced stage of the operation.

When the slag is liquid at the beginning of the operation, (as occurs in the use of borax) it is continually thrown on the sides of the scorifier, and forms a ring on the surface of the bath, leaving in the centre the metallic substance, which has a considerable extent of surface, which is continually diminishing.

The current of air being thus directly in contact with the fused metals, rapidly causes their oxidation, which does not take place when the semi-fluid substances float here and there on the metallic bath. The proportion of lead and borax necessary for a scorification, varies exceedingly according to the nature of the substance under assay, and ought to be greater in proportion as the substances, or resulting oxides, are difficult of fusion. In ordinary cases 16 parts of lead,

and 1 of glass of borax, are employed; but sometimes 32 of lead, and 3 of borax are required. A large proportion of borax is useful, especially when the substances contain much lime, oxide of zinc, or oxide of tin.

Instead of borax, glass of lead may be employed. It acts as a flux on silica; but its action is much less effective than that of borax.

There are some substances which scorify with a small proportion of lead. Thus for galena, and sulphuret of copper, 2 parts of lead suffices, but 8 parts are required for ores which contain much gangue.

Antimoniuret of silver can be scorified with 8 parts of lead; but according to experiments made in the Hartz, it appears that the slag retains about $\frac{1}{140}$th of silver, with 16 parts of lead, $\frac{1}{200}$th of fine metal is still lost; but with 3 of borax, and 16 of lead, not the slightest trace remains in the slag.

It is very difficult to separate tin and silver by the dry way. The best method is to roast the alloy in a scorifier, adding to it 16 parts of lead, and 3 of borax, at least, and operating as before described.

Speiss very nearly always contains silver, and is one of the most difficult substances to assay. If nickel be present, the button cannot be cupelled. Generally speiss may be scorified with 16 parts of lead; and the same operation is gone through twice or thrice, adding each time a fresh quantity of lead. The operation would probably succeed by roasting the speiss in the scorifier, before adding the lead.

Amalgamation.—There are a certain number of argentiferous matters, which can be assayed by amalga-

mation, as they are treated in the large way by that method. Amongst these are native silver chlorides, sulphurets, and arsenio-sulphurets, which contain neither lead or copper.

But this process is seldom had recourse to, because it is long, troublesome, and less exact than those just described.

SUBSTANCES CAPABLE OF DIRECT CUPELLATION.

Cupellation is one of the most ingenious operations that can be imagined; it has been known from time immemorial; has many characters in common with scorification, and is effected in nearly the same manner. Like that, it has for its end, the separation of silver and gold from different foreign substances, by means of lead; but it differs in this, that the scoriæ produced are absorbed by the substance of the vessel named a cupel, in which the operation is made, instead of remaining on the melted metal, so that the latter remains uncovered, and in contact with the air, so that the extraneous metals are not only oxidated, but all the lead; and there remains nothing but the pure metals, silver and gold, or an alloy of them.

Cupellation requires, as an indispensable condition, that the slag should have the property of penetrating and soaking into the body of the substance forming the cupel; it is, therefore, applicable to a certain number of substances, and not to all like scorification. The oxides of lead and bismuth, in a state of purity, are the only ores which possess the property of soaking into the cupel; but by the aid of one or the other, various oxides which by themselves form infusible

scoriæ on the cupel, acquire the property of passing through it. So that on making a cupellation, it is necessary to combine the substance with a sufficient proportion of lead or bismuth, so that the oxides they produce may combine the oxides of all the foreign metals produced in the operation, and carry them into the body of the cupel.

This proportion varies with the nature of the substances cupelled, and to other circumstances. The quantity required in ordinary cases, will be mentioned hereafter.

The cupels, or porous vessels in which the operation is made, ought to have a sufficiently loose texture to allow the fused oxides to penetrate them easily, and at the same time to possess sufficient solidity to enable them to bear handling without fracture; and, moreover, they ought to be of such a nature, as not to enter into fusion with either oxide of lead, or bismuth.

The following is the method in which an ordinary cupellation is conducted :—The furnace being heated, the bottom of the muffle is covered with cupels, placing the largest towards the end; and if they are required to be heated as quickly as possible, they may be placed topsy-turvy, and turned, at the instant of use, by means of the tongs. When the interior of the muffle is reddish white, the matters to be cupelled may be introduced. When the cupels have been placed in their proper position, from the commencement great care must be taken to blow out of them all cinders, ashes, and other extraneous substances, which may have fallen into them.

The substance to be cupelled is sometimes an alloy, which can pass without addition, and sometimes a compound, to which lead must be added. In the first case, the alloy is laid hold of by a small pair of forceps, and deposited gently in the cupel; in the second case, the substance to be cupelled is enveloped in a sheet of lead of suitable weight, and placed, as before, in the cupel; or the necessary quantity of lead may be first placed in the cupel, and when the lead is fused, the substance to be cupelled added, taking care not to agitate the melted mass, and cause loss by splashing. If the substance to be cupelled is in very small pieces, as grains or powder, it must be enveloped in a small piece of blotting paper, or still better, in a piece of very thin sheet lead, giving it slightly a spherical form, and dropping it gently into the mass of molten metal in the cupel. Sometimes the substance is gradually added, by means of a small iron spoon; but it is preferable to use paper, or thin lead, as just recommended.

When the cupels are filled, the furnace is closed, either by the door, or by pieces of lighted fuel, so that the fused metals may become of the same temperature as the muffle. When this point has been gained, air is allowed to pass into the furnace; the metallic bath is then in the state termed *uncovered*, that is, it presents a convex surface, very smooth, and without slag. When the air comes in contact with it, it becomes very lustrous, and is covered with luminous and irridescent patches, which move on the surface, and are thrown towards the sides. These spots are occasioned by the fused oxide of lead, which is con-

tinually forming, and which, covering the bath with a very thin coating of variable thickness, presents the phenomenon of coloured rings.

The fused litharge, possessing the power of moistening (so to speak) the cupel, is rapidly absorbed by it when sufficiently porous, so that the metallic alloy is covered and uncovered every instant, which establishes on its surface a continual motion from the centre to the circumference. At the same time a vapour rises from the cupels which fills the muffle, and is produced by the vapour of lead burning in the atmosphere. An annular spot is soon observed on the cupel around the metal, and this spot increases incessantly until it has reached the edges.

In proportion as the operation proceeds, the metallic bath of silver-lead diminishes, becoming more and more rounded, the shining points with which it is covered become larger, and move more rapidly; lastly, when the whole of the lead separates, the button seems agitated by a rapid movement, by which it is made to turn on its axis; it becomes very lustrous, and presents over its whole surface all the tints of the rainbow; suddenly the agitation ceases, the button becomes dull and immoveable, and after a few instants, it takes the look of pure silver. This last part in the operation of cupellation, is termed the *brightening*, *fulguration*, or *coruscation*.

If the button be taken from the muffle directly after the brightening, it may throw off portions of its substance; this must be avoided, especially when the button is not very large. The button, when covered by mammillated and crystalline asperities, is said to

have vegetated. The cause of this effect seems to be, that when the fused buttons are suddenly exposed to the cold air, the silver solidifies on the surface, whilst that in the interior remains liquid. The solid crust contracted by cooling, strongly compresses the liquid interior, which opens passages for itself, through which it passes out, and around which it solidifies, when in contact with the cool air. But it sometimes happens, that when the contraction is very strong, a small portion of the silver is thrown off in the shape of grains, which are lost.

After brightening, the cupels must be left for a few minutes in the furnace, and drawn gradually to the mouth, before they are taken out, so that the cooling may be slow and gradual. These precautions are nearly superfluous, when the buttons are not larger than the head of an ordinary pin.

As silver is sensibly volatile, it is essential, in order that the smallest possible quantity be lost, to make the cupellation at as low a temperature as may be. On the other hand, the heat ought to be sufficiently great, so that the litharge may be well fused and absorbed by the cupel; and, moreover, if the temperature be too low, the operation lasts a very long time, and the loss by volatilization will be more considerable, than if the assay had been made rapidly, at a much higher temperature.

Experience has proved that the heat is too great, when the cupels are whitish, and the metallic matter they contain can scarcely be seen, and when the fume is scarcely visible, and rises rapidly to the arch of the muffle. On the contrary, the heat is not strong

enough, when the smoke is thick and heavy, falling in the muffle, when the litharge can be seen not liquid enough to be absorbed, forming lumps and scales about the assay. When the degree of heat is suitable the cupel is red, and the fused metal very luminous and clear.

In general it is good to give a strong heat at the commencement, so as to well uncover the bath, then to cool down, and increase the heat at the end of the operation for a few minutes, in order to aid the brightening. There can be no inconvenience in urging the temperature at first, because the silver-lead is then poor, and much precious metal cannot be lost by volatilization. The increase of fire given towards the end, is for the purpose of separating the last traces of lead, from which it is very difficult to free the silver; but this strong fire must not be continued long, otherwise there might be a notable loss by volatilization. When the assay of very poor argentiferous matters is made, the heat can be kept up nearly all through the cupellation. It generally succeeds better when the temperature is too high, than too low.

The force of the current of air which passes through the muffle, is another very important thing in the success of the operation. Too strong a current cools the cupel, oxidates too rapidly, and the assay would be spoilt. With a too feeble current the operation proceeds slowly, the assay remains a long time in the fire, and much silver is lost by volatilization.

When the litharge is produced more rapidly than it can be absorbed by the cupel, or when it is not liquid enough, which may happen from the furnace

being too cold, or when other oxides, produced at the same time, diminish its fusibility, it accumulates gradually on the fluid metal, forming, at first, a ring, which envelopes its circumference, and which, extending gradually, finally covers the whole surface; at this period the assay becomes dull and all movement ceases. When the operation is carefully attended to, it is nearly always possible to avoid this accident. If, at the first moment, any signs are manifested of this evil, the temperature of the muffle must be raised, either by shutting the door, or placing in it burning fuel: the assay will, in a little time, resume its ordinary course. But when the cause of the mishap is supposed to be the abundance of foreign oxides in the assay, a fresh proportion of lead must be added.

It can be ascertained whether an assay has *passed* well by the aspect of the button. It ought to be well rounded, white, and clear, to be crystalline below, and readily detached from the cupel. When it retains least, it is brilliant below and livid above, and does not adhere at all to the cupel.

In order to detach the button, seize it with a fine strong pair of pliers and examine with a microscope, brushing it to detach small particles of litharge which may adhere to it, and place it into the pan of a balance which will indicate the $\frac{1}{1000}$th of a grain. The weight of the silver furnished by the lead or litharge employed in the operation, ought to be subtracted from the amount of silver obtained; so that it is necessary to ascertain the richness of these matters beforehand, as they are never completely free from silver. The poorest of them contain from $\frac{1}{100,000}$th to $\frac{1}{10,000}$th.

Sometimes an equal quantity of lead is placed in another cupel, and the silver thus obtained placed in the balance pan containing the weights.

Cupellation does not give the exact proportion of silver contained in an alloy. There is always a loss, and this loss is always greater than that which takes place in the large way, as in the latter process a greater quantity is always obtained than that determined by the assay. The loss of silver is traceable to three causes; 1st, volatilization; 2ndly, to oxidation, and lastly to the absorption of minute globules of silver into the body of the cupel. It is certain volatilization takes place, because a notable quantity of silver is always found deposited on the sides of the furnace and chimney in the shape of dust, and silver which is volatile by itself becomes much more so when alloyed with lead, and is carried away by the vapours of the latter, and found in the pulverulent deposits termed *lead smoke or soot* which proceeds from the combustion of the latter metal in the air. Nevertheless, this cause of loss is not very important, for it is rare that the soot contains more than $\frac{1}{10,000}$th of silver, and accurate experiments have proved that in cupellation in the small way not more than two to three per cent. of lead is volatilized. It is certain that a portion of the silver found in cupels which have been used for assays, exists in the state of oxide, for no part of their mass is free, it is found even in the bottom; besides, it is known that the carbonate of lead, precipitated from acetate of lead, made from litharge, contains silver, and a notable quantity of that metal is found even in the sulphate of lead prepared by means of alum from the acetate; for silver cannot

exist in the acetate carbonate or sulphate, but as oxide.

It has been remarked that the centres of cupels which have been used for assays, are richer in silver than the parts nearer the circumference, and that under the button there is a spot of bright yellow, which appears to be oxide of silver. But the most important cause of loss in an assay, is the property which the alloys of silver and lead possess of introducing themselves into the pores of the cupel. The quantity thus lost is in proportion to the coarseness of the cupel. For the same quantity of silver the loss which takes place in an assay, varies according to the nature of the alloy, and the circumstances under which the assay is made; so that it is not possible to form accurate tables of correction. This loss is much augmented with the quantity of lead employed, but without its being proportionate, so that when scorification is had recourse to it is advantageous to continue the operation for some length of time, in order that the metallic button may be reduced to the smallest suitable volume.

In the assay of rich alloys the proportion to the total amount of silver is very small, but notable; and it has been calculated for the alloys of copper employed in the arts at $\frac{1}{3,000}$th; but in the assay of poor ores, such as galena and other minerals treated in the large way, the loss is very great, for it is usually as high as $\frac{1}{500}$th.

By extracting the lead from cupels used in this class of assay, the metal furnished, contains from about $\frac{1}{300,000}$th to $\frac{1}{500,000}$th of silver. The following experiment will give an idea of the influence of the proportion of lead or the loss of silver: 100 of commercial

litharge were fused with 10 of black flux, and gave 27 of lead; and a slag which was pulverized and reduced in the same crucible with 15 of black flux, and a second button was produced weighing 45 parts. These two buttons being cupelled separately, gave the first 3½ and the second 1 only of silver. Three new quantities of 100 parts of the same litharge were fused; the first with ½ a part of starch, the second with 2½, and the third with 10 of the same reducing agent. The resulting buttons of lead weighed respectively, 5.28 and 79 parts. These buttons were cupelled and furnished 3½, 3½, and 3 respectively. From these experiments it will be seen that when the litharge is not reduced completely there remains a notable proportion of silver in the scoriæ; but, nevertheless, in order to extract the largest possible quantity, the whole must not be reduced. Indeed, but a twentieth part need only be reduced, because more precious metal is lost in the cupellation of a large quantity of lead than remains in the portion not reduced. The loss of silver in large cupellations is less than that which takes place in an assay, because in the large way the litharge is run off, or the greater part of it; whilst in an assay the cupel totally absorbs it, so that the latter presents, relatively to the same mass of lead, a very much smaller surface in the large than in the small way; now it can be readily seen that the quantity of silver lost by absorption into the pores of the cupel, must be proportioned to its surface, all things being equal.

It has been ascertained by experiment, that a cupel absorbs about its own weight of litharge; so that from this fact, a cupel of the proper size may be chosen,

when the weight of lead to be cupelled is ascertained. It is always better to have the cupel about $\frac{1}{3}$ or $\frac{1}{4}$ as heavy again as the lead to be cupelled.

The various metals found in an alloy, which can be submitted to cupellation, scorify in proportion to their oxidizability. Those most oxidizable scorify with the greatest rapidity, and *vice versâ*; so that those which have the greatest affinity for oxygen, accumulate in the first portions of litharge formed, which, by that means becoming less fusible, sometimes lose the property of penetrating the cupel: hence the reason why cupellations always present more difficulties at the commencement of the operation than towards the end, when the litharge formed is nearly pure oxide of lead, and which can contain only oxide of copper.

The appearance of the cupel used in an assay, will give indications of the metals the alloy contained. Pure lead colours the cupel straw-yellow, verging on lemon-yellow. Bismuth straw-yellow passing into orange-yellow. Copper gives a grey, dirty red, or brown, according to its proportion. Iron gives black scoriæ, which form at the commencement of the operation, and are generally found at the circumference of the cupel. Tin gives a grey slag. Zinc leaves a yellowish ring on the cupel, producing a very luminous flame, and occasioning losses by carrying in its vapour silver, and by projecting it from the cupel in its ebullition. Antimony and sulphate of lead in excess give litharge-yellow scoriæ, which crack the cupel; but when not produced in too great a proportion, are gradually absorbed by the litharge.

The cupellation of the alloys of copper and silver is

the most important of all, and that which is undertaken the most often. It has also been the most studied, because the results it affords are of great importance in the purchase of the precious metals. This class of cupellation is effected without difficulty, because the oxide of copper forms so slowly, that the litharge is always enabled to pass it into the body of the cupel. After having weighed the lead and placed it in the cupel, as soon as it is perfectly fused, place it in the alloy to be assayed, wrapped either in blotting-paper or thin leaf-lead. It is essential, in this class of assay, to employ a sufficient quantity of lead to carry away all the copper. We may always be sure of succeeding whatever the alloy may be, by employing the maximum proportion of lead, that is to say, the quantity necessary to pass pure copper; but as the loss which the silver undergoes increases with the length of the operation and with the mass of the oxidized matters, it is indispensable to reduce this loss as much as possible by reducing the proportion of lead to that which is strictly necessary. Long experience has proved, that silver opposes the oxidation of copper by its affinity, so that it is necessary to add a larger amount of lead in proportion to the quantity of silver present.

M. d'Arcet has obtained the following results by the most accurate experiments.

Standard of silver.	Quantity of copper alloyed.	Quantity of lead necessary.	Relation of lead to copper.
1000	0	$\frac{3}{10}$ths.	
950	50	3	60 to 1
900	100	7	70 .. 1
800	200	10	50 .. 1

Standard of silver.	Quantity of copper alloyed.	Quantity of lead necessary.	Relation of lead to copper.
700	300	12	40 to 1
600	400	14	35 .. 1
500	500	16 to 17	32 .. 1
400	600	16 .. 17	27 .. 1
300	700	16 .. 17	23 .. 1
200	800	16 .. 17	20 .. 1
100	900	16 .. 17	18 .. 1
pure copper	1000	16 .. 17	16 .. 1

It is remarkable that below the standard of 500, the same proportion of lead must be employed, whatever that of copper. This fact is repeatedly verified by experiment. Whenever fine silver is fused in a cupel, it is always necessary to add lead, in order to cause the button to unite and form well. If less than $\frac{3}{10}$ths of lead be employed, the button will be badly formed; the litharge could not separate but by the action of a very strong heat, and a considerable loss of silver would ensue. If, on the contrary, $\frac{5}{10}$ths of lead is exceeded, the cupellation goes on well, but the loss would be greater, on account of the duration of the process. These proportions also ought to vary with the temperature. M. Chaudet has found, that to cupel an alloy containing $\frac{900}{1000}$ths of silver, 5 parts of lead are required in the middle of the muffle, 10 in the front, and only 3 at the back.

The proportion of copper carried off by litharge varies not only with the temperature, but even for the same temperature in relation to the amount of copper and lead the alloy contains. By cupelling 100 parts of copper with different proportions of lead in the same furnace, M. Karsten obtained the following results:

Lead added.	Copper remaining after cupellation.	Quantity of lead consumed in carrying off 1 of copper.
100	78.75	3
200	70.12	7.1
300	60.12	7.7
400	49.40	7.9
500	38.75	8.1
600	26.25	8.15
700	19.75	8.00
800	8.75	8.70
900	5.62	9.50
1000	1.25	10.10
1050	0.00	10.50

From which we see, that the lead carried away from $\frac{1}{16}$th to $\frac{1}{10}$th of its weight of copper. Much less lead yet can be employed in a cupellation by making the alloy maintain its richness of copper throughout the operation. This can be accomplished by adding to the alloy in the cupel small doses of lead, in proportion as that first added disappears by oxidation. If, for example, an alloy composed of 4 parts of copper and 1 of silver, be fused with 10 of lead, and by adding successive small doses of the latter, as already pointed out, but 7 parts will be consumed, although in the regular way, from 16 to 17 would be employed.

The proportion of oxide of copper contained in the litharge increases each instant, and goes on incessantly increasing when an alloy of copper and lead is cupelled which it contains. According to M. Karsten, this proportion is always about 13 per cent. at the commencement, and 36, or more than a third, at the end of the operation.

In the assay of the coined alloys of copper and silver,

the loss of silver may even amount to $\frac{5}{1000}$ths; but the loss is variable, and is proportionally greater as the standard of the alloy is lower.

The following table contains the results of many experiments made on this subject.

Exact standard.	Standard found by cupellation.	Loss, or the quantity of fine metal to be added to the standard as obtained by cupellation.
1000	998.97	1.03
975	973.24	1.76
950	947.50	2.50
925	921.75	3.25
900	896.00	4.00
875	870.93	4.07
850	845.85	4.13
825	820.78	4.22
800	795.70	4.30
775	770.59	4.41
750	745.48	4.52
725	720.36	4.64
700	695.25	4.75
675	670.27	4.73
650	645.29	4.71
625	620.30	4.70
600	595.32	4.68
575	570.32	4.68
550	545.32	4.68
525	520.32	4.68
500	495.32	4.68
475	470.50	4.50
450	445.69	4.31
425	420.87	4.13
400	396.05	3.95
375	371.39	3.61
350	346.73	3.27
325	322.06	2.94

Exact standard.	Standard found by cupellation.	Loss, or the quantity of fine metal to be added to the standard as obtained by cupellation.
300	297.40	2.60
275	272.42	2.58
250	247.44	2.56
225	222.45	2.55
200	197.47	2.55
175	173.88	2.12
150	148.30	1.70
125	123.71	1.29
100	99.12	0.88
75	74.34	0.66
50	49.56	0.44
25	24.78	0.22

These numbers, however, are not constant, and vary with the circumstances under which the assays are made; two assays made from the same ingot, by the same assayer, can differ as much as four or five thousandths. Tillet has remarked that the cupels can retain double as much silver as is lost; which proves, as has already been mentioned, that the silver obtained by cupellation is not perfectly pure, but may retain as much as 1 per cent. of lead.

Sulphuret of Silver may be cupelled directly, without the addition of lead; but as it passes readily into the ordinary cupels, Schlutter remarks that the cupels for this purpose must be made of 2 parts of pulverized brick, and 1 of glass. Moreover, this means cannot be considered exact; it is much better, therefore, to make the assay in an ordinary cupel, and use lead.

Argentiferous Galenas may be cupelled directly, whenever they are nearly pure, or contain but 2 to 3 per cent. of foreign matters, such as sulphate of baryta,

pyrites, &c., or a much larger proportion of quartz. It is possible, therefore, to make this cupellation without addition, but it is then a true roasting at a high temperature, and not to be much recommended.

From all that has been stated concerning the operation of cupellation, it will be seen, that although it is the one generally resorted to, it is far from exact; and indeed, when conducted with every possible care, is but an approximation to the truth; so that much modification, or indeed complete alteration is required in the assay of argentiferous matters. There is, in truth, no exactitude to be attained, excepting by the use of the wet method of assay or analysis. Gay-Lussac has paid much attention to this matter, and has contrived various apparatus to facilitate the operation. His method consists in determining the standard of argentiferous substances, by means of a standard solution of common salt, a certain amount of which will be required to precipitate a certain amount of silver in a given weight of alloy. This process is founded on the following principle. The alloy previously dissolved in nitric acid is mixed with a standard solution of common salt, which precipitates the silver as chloride, a compound perfectly insoluble in water, and even in an excess of nitric acid. The quantity of chloride of silver precipitated is determined, not by its weight, a process which would involve too much time, but by the weight or volume of the exact amount of standard solution of salt necessary to precipitate the whole of the silver contained in the alloy operated on.

The complete precipitation of the whole of the silver can be readily determined by the absence of all cloudi-

ness in the solution of the alloy on the addition of a small portion of the standard solution of salt; a cloudiness will take place, even when half-a-thousandth of metal remains in the solution. By agitating briskly the liquid, which is cloudy by the presence of chloride of silver, for a few minutes, it will become perfectly bright. The presence of lead and copper, or any other metal except mercury, has no perceptible influence on the quantity of salt necessary to precipitate the silver; that is to say, the same quantity of silver, pure or alloyed, requires for its precipitation a constant quantity of the sea-salt. When mercury is present, a cupel assay must be had recourse to.

The method, as proposed by Gay-Lussac, requires many little apparatus which are expensive, and do not, in my opinion, give very much accuracy to the process. I employ, in this class of assay, very simple apparatus, similar to those used in alkalimetry, to determine the weight of the standard solution of salt added; and the results so obtained always correspond.

The apparatus I employ are as follows: a small flask for the solution of the silver to be assayed, a stoppered bottle (containing from 3 to 4 fluid ounces), in which the decomposition of the silver solution is made, and two small alkalimeters, known by the name of Schuster's.* The alkalimeter is a light glass bottle, with two openings, one of which is drawn out, and extends over the side of the flask, parallel to its bottom; this aperture is for the purpose of allowing the fluid contained in the bottle to pass out in single drops, which it

* They may be obtained at any philosophical apparatus warehouse.

most effectually does. The other aperture just mentioned, is furnished with a small stopper, and is used for the introduction of the fluid. An accurate balance and weights, with a few stirring rods, complete the set.

The standard solution of salt is made as follows: It is absolutely necessary to employ pure salt; it is better to manufacture this, which may be accomplished by accurately neutralizing pure hydrochloric acid with bicarbonate of soda, evaporating the solution to dryness, and fusing the dry residue, taking care to place it, whilst warm, in a well-stopped bottle, to preserve it perfectly free from moisture.

Distilled water must also be employed, of which 94.573 parts must have added to them 5.427 parts of the salt, as above prepared. The solution so formed must be kept in glass stoppered bottles, and exposed as little as possible to the air during manipulation, otherwise it will become sensibly stronger by evaporation, and cause fallacious results; 100 grains, by weight, of this solution, precipitate exactly 10 grains of silver.

I also employ another solution, the use of which will be pointed out hereafter. This solution is made by dissolving 10 grains of pure silver in a small quantity of nitric acid, and adding pure water (distilled) to the solution, until its weight amounts to 10,000 grs. This is the *verifying* solution.

This solution must be preserved with the same precautions as to exposure to air, &c., as the last; in addition to which, it must be kept in a dark place.

The assay is thus made: 10 grains of the alloy are dissolved in nitric acid; when the solution is effected, water (distilled) must be added, and the whole poured

into the assay bottle before mentioned. The flask in which the solution was made must be carefully washed out, and the rinsings added to that already in the assay bottle. A quantity (any amount) of the solution of salt must be placed in one of the alkalimeters, which, with it, must be carefully weighed, and the weight noted. The standard solution is now to be added, drop by drop, to the solution of the alloy in the bottle, replacing the stopper (taking care to hold it in the hand whilst dropping in the solution of salt) after each addition, and shaking the bottle well, to clarify its contents; repeating the above routine, until the last drop occasions no turbidity in the liquid.

The weight of the alkalimeter and contents must now be again taken, and the amount of grains of salt solution employed, noted.

There is most likely now in the bottle a little excess of salt, the amount of which must be estimated by the verifying solution, just mentioned, in the following manner. Place in the other alkalimeter a certain amount (any quantity) of the verifying solution, and ascertain its weight with that of the alkalimeter, taking care to note it. Now add the solution, drop by drop, to the assay in the bottle, observing all the precautions as to agitation, &c., already pointed out, until the last drop causes no turbidity; then weigh the alkalimeter, and note the loss of weight, and from the amounts of solution used, calculate the standard of the silver alloy in thousandths. This will be rendered perfectly clear by an example: 10 grs. of alloy require for complete precipitation, 60.7 grs. of the salt solution; and as 10 grs. of the solution are equal to 1 gr. of silver, 60.7 is equal

to 6.07 of silver; but a slight excess of solution has been added, which has increased the amount of silver above its true quantity, therefore, 52 grs. of the verifying solution were added, and as each 100 grs. of such solution contains .1 of a grain of silver, the 52 grs. will contain .052 of silver, which, deducted from 6.07=6.018 of silver, which gives 601.8 thousandths as the true standard of the alloy operated upon.

I conceive, by my method of operation as described above, as accurate results can be obtained as by the apparatus employed by Gay-Lussac, and in a shorter time than either by his process or by the cupel; the method is far more exact than that of the cupel, and much cheaper in operation.

CHAPTER XVI.

ASSAY OF GOLD.

AURIFEROUS matters are thus distinguished.—First, Those which cannot pass the cupel directly; they are for the most part ores—some alloys. Secondly, Those which pass the cupel; they are some minerals, such as the galenas, tellurets, &c., and the alloys with the metals which can be separated from silver by this process. Thirdly, The alloys with silver and platinum, which cannot be assayed with exactitude, excepting by the *humid way*.

AURIFEROUS MATTERS WHICH DO NOT PASS THE CUPEL.

General Method.—These substances are assayed in exactly the same manner as analogous argentiferous substances, by fusion after roasting, or without roasting, or by scorification. As these bodies are always extremely poor, it is necessary to take 400 grs. at least, instead of 200 grs., which quantity of silver ore is sufficient. Generally when they contain oxide of lead they are fused with black flux, when they contain only oxidated substances, but not oxide of lead, they are fused with black flux and litharge; when they are

essentially composed of stony gangue mixed with oxidizable substances, such as arsenical or cupreous pyrites, which is most frequently the case, they are fused with litharge only; and lastly, when they are nearly pure oxidizable substances, they are fused with a mixture of litharge and nitre, in order not to be compelled to cupel a large quantity of lead. It is essential that the mixtures be perfectly fused, in order that no metallic grains be left in the scoriæ; and, as the mass to be fused is often large, it is better to divide it into two or three portions and treat them separately in crucibles just large enough to contain each portion when three-fourths full. When the matters to be assayed contain sulphur, it is extremely important that the operation be conducted in such a manner that it is all burnt off, and especially as alkaline sulphurets are formed; because these sulphurets hold in combination a certain quantity of gold, which neither lead or any other metal can deprive them of.

The example we shall cite will show how great the loss is that can be occasioned in this way :—

10 grammes = 154 grs. of the *sulpho-plumbiferous telluret of gold of Nagyag* perfectly pure, and in which was found by the humid way 0.067 of gold was fused with 30 grammes = 462 grs. of black flux, and produce a button of ductile lead weighing 5.2 grammes = 80 grs. and a compact scoriæ of the colour of argol. The scoriæ was acted on with water; the liquor became slightly yellow and gave a saffron coloured precipitate, with disengagement of sulphuretted hydrogen. The insoluble residue being fused with 10 grammes = 154 grs. of the same flux gave a brittle button of lead

weighing 0.9 grammes = 13.95 grs., and a new scoriæ similar to the first. This scoriæ was treated with water, and the liquid was exposed to the air until completely decolorized; and by treating the residue with nitro-hydrochloric acid, &c., it was found to contain a very notable quantity of gold. The two buttons of lead were cupelled, the first passed well without the addition of lead, (but there remained on the cupel a scoriaceous orange border produced by antimony) and left a button of gold weighing 0.4 grammes = 6.16 grs. The second contained tellurium, and could not pass without the aid of a large quantity of lead, and left .15 grammes = 2.32 grs. of gold. Thus in all, there was obtained but .55 grammes = 8.47 grs. of gold, from 10 grammes = 154 grs. of a mineral, which contained .67 grammes = 10.318 grs.

Iron.—10 grammes = 154 grs. of the same mineral fused with 2 grammes of iron filings = 30.8 grs., gave a very brittle button of lead weighing 5.6 grammes = 86.24 grs. which cupelled with difficulty, and gave a metallic crystalline scoria of a deep blackish green colour. This scoria was fused with 150 grammes = 23.16 grs. of litharge, a button of lead was the result, which gave by cupellation .07 gramme = 1.078 grs. of gold; the sulphurous scoriæ had then retained the eleventh of the gold contained in the mineral.

Litharge.—The mineral can be assayed by fusing it with 8 and even with 4 parts of litharge, and the scoriæ contain but a very small quantity of gold; nevertheless the button is difficult to cupel because it contains much antimony and tellurium, and produces much scoriæ. In order to obtain an exact result, it is better to employ

20 or even 25 parts of litharge; then the lead retains but very minute quantities of antimony, and contains but a trace of tellurium, and the scoriæ do not contain any gold.

There are other methods of assaying the ore of Nagyag, which we will give in detail, because they are useful for the extraction of gold and tellurium from this ore with economy.

Nitre.—The metallic matter well separated, by washing, from the gangue, is heated with its own weight of carbonate of soda, and from .8 to .9 of nitre. There is much boiling, but no deflagration or bubbling over. When the fusion is complete, the whole is poured into an ingot mould and pulverized. The powder can be used instead of carbonate of soda in a fresh operation, in order to temper the action of the nitre on the mineral, employing always 80 or 90 of nitre to 100 of the mineral. When the whole quantity on which we wish to operate has been treated in this manner, it is left to cool in the crucible, after having given it a strong fire for a few minutes. Then break the crucible and a metallic button is obtained, the weight of which is from .12 to .15 of that of the mineral, and contains all the gold combined with lead, antimony, and a little tellurium. Besides this button, there is found some fused litharge slightly brown; and, lastly, a saline opaque scoria of a whitish colour, approaching to a fresh salmon yellow. This scoria is composed of carbonates, sulphates, and tellurets of potash and soda, with a small quantity of an alkaline antimoniate, and is coloured by a little disseminated litharge. It is to be bruised and digested in a large quantity of water, and filtered, which

separates the oxide of lead. Then supersaturate the liquid with sulphuric acid, filter afresh, in order to separate a small residue which is not dissolved, and which is composed of antimony, silica, and a little tellurium; lastly, the tellurium is precipitated from the solution by means of plates of soft iron well cleansed from rust. It is then washed with a little hydrochloric acid, in order to free it from any remaining particles of iron, and is then perfectly pure. As for the gold, it can be extracted from the metallic button by the *dry way*, scorifying it with litharge, or even cupelling it with lead. If it is wished to proceed by the humid way, it is first treated with pure nitric acid, which dissolves the lead, tellurium, and oxide of antimony. Then after a good washing it is boiled with concentrated hydrochloric acid, washed, and the gold remains pure. If, in order to act on the mineral, 1 part of nitre be used instead of .8 or .9, the gold would be entirely disengaged from the compound, but it would not form into an even button, and would remain disseminated in grains in the litharge; and in order to extract it without loss, lead must be thrown into the fused matter in the crucible—heated in a strong fire, and the button obtained cupelled. If, on the contrary, we add to the mineral but .36 to .40 parts of nitre, the sulphur alone will be burnt, and a button of tellurets of gold, lead, and antimony, will be obtained, weighing about .80 parts. By treating this button with pure nitric acid all the lead and tellurium are dissolved, and the residue contains metallic gold and oxide of antimony, which can be separated from the gold by means of hydrochloric acid.

Sal-ammoniac.—By heating the mineral of Nagyag in

a crucible with 2½ parts of sal-ammoniac, hydrosulphate of ammonia is disengaged, which carries away the little sulphuret of antimony, and a button of telluret of gold perfectly pure remains, the weight of which is .20; it is enveloped in a grey scoriæ of chlorosulphate of lead and antimony.

Chloride of Lead.—The telluret of gold can be separated from the ore of Nagyag by fusion, with 3 parts of chloride of lead, which forms a scoriæ with the sulphurets; but it is possible for the tellurium to retain a small quantity of antimony.

We should succeed, probably, in obtaining economically a pure telluret, by employing a mixture of 3 parts of chloride of lead and one of sal-ammoniac.

Alkaline Sulphurets.—The ancient assayists took advantage of the property which the alkaline sulphurets possess, of dissolving the sulphuret of gold, in assaying the auriferous pyrites; but the processes founded on this property are much less exact and convenient than those to be presently pointed out. Schlutter mixed the auriferous pyrites with 3 parts of carbonate of potash, and threw the mixture gradually into a red hot crucible. It was then fused rapidly. The fused matter was powdered, boiled in water, and filtered; all the gold contained in the mineral, according to him, is dissolved in the liquid. In order to separate it, the liquid was saturated with acetic acid. The sulphuret of gold was deposited, mixed with a large quantity of sulphur, from which it was easy to separate the pure gold by roasting it in a cupel, and adding a little lead towards the end in order to reduce it to a button, and fine it; others have detonated this sulphuret with nitre

in a crucible, and adding grains of lead, fused it, and cupelled the button.

Nitric Acid.—Sage treated auriferous pyrites with 12 parts of pure nitric acid, collecting the residue in which all the gold is found, scorifying it with lead, and then cupelling.

Roasting and Washing.—M. Boussingault assures us that auriferous pyrites can be assayed with much exactitude by roasting them completely, by which they are changed into a very light oxide of iron, and carefully washing the roasted matter, the gold which exists in the pyrites in the metallic state remains in the washing apparatus.

ALLOYS; OR, MIXTURES OF GOLD WITH IRON, TIN, ZINC, BRONZE, OR BRASS.

These alloys, or mixtures, proceed from worn out gilt ornaments. In order to recover the gold which is placed on their surface, so that it be mixed with the least possible quantity of the metal of which they are made, they are rasped with a rasp, or file, or treated by different chemical processes, which remove the gold by dissolving it, or which detach it by dissolving the metal to which it adhered. We can, for instance, cover the gilt object with sal-ammoniac moistened with nitric acid, and heat to dryness, or rub it over with oil, and sprinkle it with a mixture of two parts of sal-ammoniac and one of nitre, and heat it. After one or the other of these operations the gold is detached by a blow, or by simple friction with the scratch brush.

The different mixtures of gold and iron can be assayed, by fusing them with litharge or with nitre, and

adding lead after fusion to collect together all the particles of metal, or by submitting them to scorification with about 16 parts of lead and a certain quantity of borax. When this last process is followed, it is indispensable to push the operation so far, that neither tin nor zinc remain in the lead; because these metals would ruin the cupellation; because the latter would produce, by its rapid evaporation, losses by particles being projected.

Sulphur and Potash.—In order to separate gold from iron, the old assayers recommended very different methods. Some fused with sulphur and three of potash. They washed in water, which dissolved the sulphuret of gold, precipitated the sulphuret by an acid, and separated the gold by the means already pointed out.

Sulphur and Copper.—Others, again, fused with one part of copper and two of sulphur. Then roasted the sulphuret, and treated with nitric acid, and fused the oxidized matter with litharge, black flux, &c. Copper having a very great affinity for gold, is one of the most effective agents for separating gold from its combination in the *dry way*. Others again employed the humid way, dissolving in *aqua regia* and precipitating the gold by protosulphate of iron.

Sulphuret of Antimony.—Gold may be separated from iron, tin, zinc, and by means of sulphuret of antimony. This process cannot be well applied in assays, but is employed by goldsmiths to bring gold to a very high standard, by taking from it the least traces of different metals, even silver, which can be retained after cupellation and after parting. The operation is executed thus: the gold is heated in a crucible, and when fused very pure sulphuret of antimony is thrown on it, in the

proportion of 2 to 4 parts, according to the more or less abundant presence of foreign metals. It is heated moderately in order to avoid spirting over, and taking care that no cinder falls into the metallic mass while in a state of fusion; because it would then exhibit a lively effervescence, by which the contents of the crucible might run over the sides. In order to avoid all chance of loss, it should be of such a size that when the whole mass is in a state of fusion it should be but two thirds full. All foreign metals, even silver, pass into the state of sulphuret, and the antimony left by the sulphur alloys with the gold. The fused mixture is to be poured into a conical iron ingot mould. The sulphurets and antimoniuret of gold are separated and fused with a fresh quantity of sulphuret of antimony, and the operation repeated as many times as judged necessary to complete the purification. When the proportion of foreign metals combined or mixed with the gold is considerable, sulphur is often added to the sulphuret of antimony in the first treatment.

Gold and Antimony, Nitre.—The antimony must now be separated from the gold. Three processes can be employed for this purpose; the alloy may be fused with nitre, and then blown upon by a pair of bellows until pure gold is left. When nitre is used, the alloy is placed in a crucible, with three parts of that salt, covering it with another crucible reversed, in the bottom of which is pierced a hole, which can be closed at pleasure with a plug of clay, and it is to be gradually heated, applying heat to the upper part first, in order to avoid bubbling over, then towards the end employ a strong fire. The operation goes on well

when the bubbling in the crucible is small and regular. As long as undecomposed nitre remains, a lighted coal, when placed near the hole in the crucible, burns brilliantly. When this effect ceases to be produced, a strong fire may be employed without fear of loss by spirting.

Roasting.—In order to separate gold and antimony by simple roasting, the alloy must be fused in a crucible, and when well heated, the blast from a pair of bellows must be thrown upon the mass. The blowing must at first be conducted cautiously, because if the antimony volatilized very rapidly, it might carry away a little gold, (Macquer, who described this operation, remarked that a current of air, continually kept up, much augmented and favoured evaporation). The current of air must be just so great as to produce a smoke. When the gold approaches a state of purity, it seems to coagulate or thicken, and becomes covered with a species of skin; the heat is then augmented, and the crucible corked, then uncorked and blown into for a few minutes; lastly, a strong fire is employed to drive off the last particles of antimony. Often, in order to have the gold of the highest standard possible, it is fused with a fourth to a fifth its weight of nitre. When perfectly pure, its surface is without clouds, and quite brilliant.

If it is nothing but an assay of an alloy of gold and antimony that has to be made, it can be fused with litharge, or nitre, or scorification, or even direct cupellation, with or without the addition of lead.

Galena.—Galena, either pure or mixed with sulphur, can be used like sulphuret of antimony to separate

all the metals alloyed with gold; the latter alone remains alloyed with the lead, which can be separated from it by cupellation, but the gold thus obtained would retain silver, and not be so pure as that obtained by sulphuret of antimony.

Peroxide of Manganese.—According to Mr. Andrew Thompson, we can separate gold from its alloys with all other metals except silver by treating them with peroxide of manganese. The alloy is pulverized or reduced to fine leaves by the flatting mill, which leaves, when malleable, are coiled into a spiral form. It is then mixed with peroxide of manganese, and heated for a quarter of an hour at the fusing point of gold; 3 parts of powdered glass are then mixed with the whole, and fused; the gold is reduced to a button, and the foreign metals, separated in the state of oxides, remain dissolved in the scoriæ. The silver of the alloy contained in it is best part found with the gold; but there is a portion, which is sometimes considerable, becomes oxidized and remains in the scoriæ. This operation can be simplified and rendered easier by mixing at once the pounded glass alloy and a little litharge with the peroxide of manganese, and fusing at once.

Amalgamation.—All that has been said concerning the amalgamation of silver ores is applicable to those of gold. This process can be advantageously employed to collect the native gold disseminated in a large quantity of earthy or other matter, on which mercury does not act.

In the valley of d'Anzasca, (Italy), principally at Macugnagna and Vinzone, are found a multitude of

small threads of auriferous pyrites, which traverse the gneiss. The richness of these pyrites varies from 0.00001 to 0.00050. They are worked in small factories, whose number amount to more than two hundred. After the pyrites are picked, they are ground in horizontal mills until reduced to grains about the size of a pea; they are then amalgamated without roasting by being turned twenty-four hours in the mills with mercury and water. The mercury is then distilled in retorts of iron. The loss of mercury is calculated at one fourth of the quantity employed.

AURIFEROUS MATTERS WHICH CAN BE DIRECTLY CUPELLED.

These are firstly: All ores analogous to those of silver which can be cupelled directly. Secondly, alloys with lead, antimony, mercury and copper.

There is nothing to add relative to those ores which have been spoken of under silver, and we have but few observations to make concerning these alloys.

Gold and Lead, Cupellation.—The cupellation of the alloys of gold and lead are conducted in a similar manner to those of silver and lead. It presents even less difficulty, and requires less precaution because it is not so volatile, and because it has a less tendency than silver to penetrate into the cupel, and the button is less subject to throw pieces out of the cupel. These cupellations take place at a higher temperature than those of silver, and we need not be afraid of giving a good heat at the moment of brightening: the gold is but the purer.

Gold and Copper, Proportion of Lead.—The alloys

of gold and copper are cupelled like the alloys of gold and silver; but as copper has a very great affinity for gold, it is necessary to use a larger proportion of lead to ensure its oxidation when combined with gold than when united with silver. This proportion varies according to the standard and the temperature. It is admitted that for the same standard, there must, under similar circumstances, be twice as much lead used in the cupellation of gold as for that of silver. Thus, 14 parts, at least, ought to be employed in common furnaces for an assay of gold coin which contains 0.100 of copper. There is no inconvenience in employing a little more, as it does not increase the loss of gold. However great the proportion of lead may be that is added to the cupreous gold for the purpose of cupellation, the button retains always a very small quantity of copper, which a fresh cupellation does not free it from, and which occasions what is termed the surcharge. This surcharge being very slight, can be neglected in assays of minerals, but it is necessary to take notice of it in the assay of alloys. But it is known that the presence of silver much facilitates the separation of copper from gold, and as it is rare that an alloy of cupreous gold does not contain a little silver, which must be separated, and when that is not the case, a small quantity of that metal can be introduced into the alloy, so as to be in about the proportion of 3 parts to 1 of gold. When an assay is to be made of an alloy of gold and copper, a sufficient quantity of silver to fulfil this condition according to the presumed standard, which is determined approxima-

tively by a preliminary assay, and then cupelled with lead.

Treatment in the large way.—In the large way, gold cannot be extracted from a very poor alloy of copper, because it would occasion a very large consumption of lead in relation to the quantity of gold obtained. The operation is commenced by separating the greater part of the copper by means of sulphur, iron pyrites, or galena, the rich alloy is then cupelled; if the matt contains gold it must undergo a roasting, and then be fused; by these means the black auriferous copper is separated from the impoverished matt.

Touchstone.—It is often sufficient in commerce to determine approximatively the standard of alloys of gold and copper by the proof of the touchstone. The following is the mode of operation: Make on the touchstone a trace with the alloy; then rub this trace with the feather of a pen, moistened in the acid liquid composed as described further on. This acid dissolves the copper; the effect is then examined, after which wipe it slightly to take away the liquid, and examine afresh that which remains of the trace. When in the habit the standard of the alloy can be ascertained very nearly according to the more or less green tint the acid assumes, and the thickness of the trace of pure gold which remains on the stone. In order to accustom oneself to this proof, we must make comparative assays with different known alloys, moulded into the form of strong needles, known by the name of touch needles, and it can be noticed with which of these needles the alloy is identical.

The Acid Liquid.—The liquid which is used to act on the metallic streak is composed of 98 parts of nitric acid of the density of 1.340, two parts of muriatic acid of a density of 1.173, and 25 parts of distilled water. The composition of this liquid has been determined by trial, and at the temperature of 50^0 to 54^0, at which we ought always to operate; it is without action on alloys of the standard of 0.750, and above. The alloys of an inferior standard are attacked by this acid liquid; they become brown, and the liquid becomes green, and when the liquid is removed, there remains a metallic trace, smaller in proportion to the quantity of copper contained in the alloy. If we operate at a temperature lower than 50^0, the acid does not attack all the alloys of a lower standard than 0.750. On the contrary, if the temperature was more elevated, the acid would attack the alloys of .750, and a little above. When assays are made in unknown circumstances, we commence by trying the effect of the acid of the traces left by needles of a known standard.

Gold, Silver, Platinum, and Copper.—The presence of platinum in an alloy, renders the separation of the oxidizable metals, more especially copper, very difficult by cupellation. It appears, indeed, that it would be almost impossible to arrive at it, if the alloy of copper contained nothing but gold and platinum. It is necessary that silver be present at the same time. When this metal is absent, it is requisite to add a quantity of it, which ought to be equivalent to double the weight of the gold and platinum united, and cupel at the strongest heat which can be obtained in a good

muffle with a suitable proportion of lead. This proportion varies much, according to the composition of the alloy, and the temperature at which the operation is carried on.

Experience has shown that the copper can be more completely separated, and less silver lost, by cupelling at a high temperature, with the least possible quantity of lead, than by employing more lead, and working at a lower temperature. M. Chaudet has made several assays, in order to determine the proportion of lead required for the cupellation of the three following alloys:

	1	2	3
Gold . .	0.100	0.020	0.005
Platinum .	0.100	0.200	0.300
Silver . .	0.250	0.580	0.595
Copper .	0.550	0.200	0.100

And has found for the first, that by employing 20 parts of lead, the separation is very nearly complete; but that at a higher temperature, there is a loss of silver; and in order to render the assay correct, it must be cupelled at the latter temperature, with only 14 of lead; for the second, 8 of lead, at a high temperature; and for the third, 30 parts of lead are necessary, at the same high temperature of the muffle; but it is almost impossible to separate all the copper, and no advantage can be obtained by increasing the quantity of lead. When almost the last traces of the copper are separated, the button must be cupelled afresh, with a small quantity of lead; but a small quantity of silver is nearly always lost. In all cases, in order that no lead shall remain, it is necessary

to leave the assay button some few minutes in the muffle, after the cupellation is finished.

The alloys of gold and silver which contain platinum, show either by cupellation or parting, certain characters which prove the presence of that metal. If the assay be not heated very strongly, it does not pass, and the button becomes flat; this effect becomes very sensible, when the platinum is to the gold, as the proportion of 2 to 100. Under the same circumstances, the nitric acid solution proceeding from the parting, is coloured straw-yellow. At the moment an assay of an alloy containing platinum terminates, the motion is slower, and the coloured bands are less numerous, more obscure, and remain a much longer time, than when there is no platinum; the button does not uncover, and the surface does not become as brilliant as that of an alloy of gold or silver, but it remains dull and tarnished. When the assay is well made, it is to be remarked that the edges of the button are thicker, and more rounded than in ordinary assays, and it is of a dull white, approaching a little to the yellow; and lastly, its surface is wholly, or in part crystalline. These effects are sensible, even when the gold does not contain more than 0.01 of platinum. When the alloy contains more than 10 parts of platinum to 90 of gold, the annealed cornet produced in the parting process, is of a pale yellow, or tarnished silver colour.

Gold alloyed with Silver.—The separation of gold from silver, is termed *parting*. Parting can be made an analytical process, when operating in the *wet way*. In the large scale it is executed sometimes in the

dry way, according to the various methods to be presently pointed out.

Parting is not only used to separate silver from gold, but for the separation of other metals, such as copper, when cupellation does not separate it entirely. Parting by the wet process, is carried on by the means of nitric acid, aqua regia, or sulphuric acid.

When an alloy of gold and silver has been reduced at the flatting mills to very thin plates, it is sufficient that it contains 2½ of silver, to 1 of gold, in order that the parting may be effected completely by nitric acid, and takes place much less easily, when the silver in the alloy is in larger proportion; but when this proportion exceeds 3 parts of silver for 1 of gold, then the latter is obtained in leaves so fine, that there is risk incurred of losing some in the subsequent manipulation, and even by the act of boiling the acid liquid.

We must always, therefore, when a very exact assay is required, contrive that the alloy shall contain a little less than 3 parts of silver to one of gold: a proportion which long experience has demonstrated to be the best. If the alloy contain less than 2½ of silver, to 1 of gold, the silver does not wholly dissolve, because there is a part of it so enveloped in the gold, that the strongest acid does not act on it.

Inquartation.—The operation by which the alloy is brought to this standard, is termed *quartation*, or *inquartation*. It consists in fusing the alloy in a cupel, with 2 parts of lead, and the quantity of fine silver, or fine gold, necessary to bring it to the desired composition. This quantity is estimated according to the

approximative determination of the standard of the alloy, which ought to be made either by means of an analytical assay, or by means of the touchstone. In order to make assays by the touchstone, touch-needles of various well known standards must be provided. When an alloy is to be assayed, it must be rubbed on the stone, and a needle formed which shall leave a trace of the same colour. If we do not employ the whole of the alloy, the assay will not be exact, because the gold and silver are not always found distributed in an uniform manner, at least, every time it is not poured into a cold ingot mould.

Operation.—The cupelled and quartated button, is flattened on an anvil, and annealed, in order to soften it. It is laminated to give it a certain thickness, and is then annealed afresh, and rolled into a cornet, or spiral, around the quill of a pen. It is necessary that the alloy should be reduced to a suitable thickness, on the one hand, in order that the silver may be dissolved completely, and on the other, that the plate of gold may remain whole after the operation. The following is that which experience has proved best. The quantity of matter operated upon, or taken for the assay, should be about 12 grains, and the alloy resulting from these 12 grains, and the silver employed in the inquartation into a plate of from 18 to 20 lines in length, and 4 or 5 in breadth.

The cornet for assay is placed in a glass mattrass, capable of containing about 3 ounces of water, and we pour on it, at different times, pure nitric acid, and heat; when all the silver is dissolved it is washed by decantation with water, then the mattrass is re-

versed into a small crucible in order to make the cornet fall out and dry it. In this state the cornet is very fragile, and of a dull red colour; it is annealed in a muffle, and heated gradually without fusion. It becomes thereby much contracted, and acquires a metallic lustre, and so much solidity that it can be weighed without fear of breaking it. Its weight can be ascertained in an assay balance.

There are many ways of employing nitric acid. Formerly, 2½ ounces (thirty-five times the weight of the alloy) of nitric acid at 20° (1.15 sp. gr.) was poured upon the inquartated cornet, and boiled gently for fifteen or twenty minutes, then decanting the liquid, and replacing it by 1½ of acid at 30° (1.24), or 32° (1.26), (twenty-four times the weight of the alloy), boiling for twelve minutes, then decanting and washing, &c. Vauquelain pointed out in his "*Manuel de l'Essayeur*" to pour on the quartated cornet—the weight of the assay being 0.5 gra. = 7.7 grains 36 = 554 grains to 50 = 770 grains of nitric acid at 22° = 1.16 sp. gr. which ought to fill the mattrass half or two thirds, and boil gently for twenty, or twenty-two minutes at most, to decant and replace the liquid by 30 to 50 grammes of acid at 32° = 1.26 sp. gr. and to boil for eight or ten minutes. The assay is to be acted on always twice, because if we employ at once very strong acid, the action will be too brisk, and the cornet might be broken or carried out of the mattrass, and on the other side, the acid of 22° = 1.16 sp. gr. cannot dissolve the last portions of silver, which are very difficult to separate from the gold.

Surcharge.—It is remarked that by following this

method the cornet always retains a small quantity of silver, so that fine gold submitted to quartation and parting, always weighs more after than before the operation. The augmentation of weight which it undergoes is termed the *surcharge*; this surcharge is commonly from 0.001 to 0.002. M. Chaudet has found means to avoid it. In order to do so, pour on to the quartated cornet nitric acid of $22^0 = 1.16$ sp. gr. and heat for three or four minutes only, replace this acid by acid at $32^0 = 1.26$ sp. gr., and boil during ten minutes, decant and make a second boiling with acid at $32^0 = 1.26$ sp. gr., which boil for eight or ten minutes. The assay requires but from twenty to twenty-three minutes; and according to M. Chaudet, gives perfectly pure gold.

Parting, on the large scale, can also be effected by nitric acid, but then 4 parts of silver instead of 3 to 1 of gold must be introduced into the alloy. It appears, according to M. Chaudet, that if we proceed as above indicated, all the silver may be separated by one action of acid at $32^0 = 1.26$ sp. gr.; but if the alloy has a higher standard, two boilings are necessary.

Argentiferous and Auriferous Ores.—In the assay of auriferous ores, the button produced by cupellation commonly contains silver. When the proportion of this metal surpasses that of inquartation, the button is flattened between two pieces of paper, and treated by pure nitric acid. The gold remains under the form of a yellowish-brown powder, which is weighed immediately, or fused at the cupel enveloped in a sheet of lead. When the quantity is extremely small and imponderable, we can assure ourselves at least of its presence

by treating the residue left by nitric acid with aqua regia; if it contain gold, it dissolves and gives a yellowish liquid, in which a drop of solution of chloride of tin or the crystallized chloride forms a deposit of purple of Cassius of a violet colour; this character proves the presence of the smallest traces of gold. When the gold predominates in the button, it is necessary to re-fuse it with three times or less its weight of silver, and recommence the assay with the addition of this preparation of silver.

Sea-salt.—We can, according to M. Gay-Lussac, make assays of the alloys of gold, silver, and copper, with great exactitude, by means of a standard solution of sea-salt. When the alloy contains five or six times more of silver and copper than of gold, a known weight of the alloy is taken, containing nearly 1 gramme = 15.4 grs. of silver; it is dissolved in a mattrass capable of containing about 200 grammes = 3080 grs. of water, with 30 grammes = 462 grs. of nitric acid, at 32^0 = 1.26 sp. gr., and boiled for ten minutes. The assay is finished as usual; but in order to leave the gold and separate the silver, supersaturate the solution with ammonia, which dissolves the chloride; wash the residue twice in succession with ammonia, then place in a crucible to anneal. If the gold were alloyed with silver and copper in a larger proportion than 1 to 6, a known quantity of fine silver should be added, and then deducted from the assay. In order to avoid all loss, the bottom of the crucible is lined with paper, and the alloy placed thereon, and the latter covered with fused borax.

Aqua Regia.—When gold is the largest portion of

the alloy, and when there are reasons for not adding silver, the parting can be made by aqua regia. In this case, all the gold is dissolved, and the silver converted into chloride; the chloride is washed, dried perfectly, and weighed. When the gold is precipitated by protosulphate of iron, it is washed with a little muriatic acid, and annealed strongly before weighing or even carrying the annealing so far as to fuse it, and then cupelling it with lead.

If an alloy, containing much silver, be treated by this process, it sometimes happens that the excess of chloride of silver prevents the complete solution of the gold. In this case it is necessary to reduce the alloy to an excessively thin plate, to dissolve the chloride in ammonia, and to treat afresh with aqua regia. This process can rarely be made use of in the large scale, because the precipitation of gold by sulphate of iron is long and troublesome.

Method of M. Rose.—M. G. Rose fuses the alloy with lead, over a spirit-lamp, in a porcelain crucible, acts on it with nitric acid, which dissolves the silver and lead, precipitates the silver by a solution of chloride of lead; lastly, the auriferous residue is dissolved by aqua regia, and the gold precipitated by protochloride of iron.

Sulphuric Acid.—The parting by means of concentrated sulphuric acid was practiced by some assayers in the time of Schlutter. It has the advantage of giving pure gold without surcharge, but is difficult to execute, and requires many precautions, on account of the high temperature which is necessary to boil sulphuric acid, and on account of the starting and bumping

which results. Besides, it is necessary to employ a very large excess of acid to hold the sulphate of silver which is formed in solution, and which, as is well known, is only very slightly soluble in water. The alloy ought to undergo quartation as for nitric acid. This process is rarely used as a means of assay, but is employed nearly exclusively to extract gold from silver, on the large scale, more especially in France, where sulphuric acid is very cheap, and nitric acid extremely dear. The following is the process: If the alloy contain oxidizable metals, such as lead and tin, and in a large proportion, they are separated by cupellation. If their quantity be very small, they can be separated, as when copper is in excess, by an operation with nitre. Long practice has made known that the alloy which is best to act upon in the large way, consists of:

Silver	0.725
Gold	0.200
Copper	0.075

It ought never to contain more gold, because then all the silver would not be acted upon; and not more copper, because the sulphate of that metal which is formed being insoluble in concentrated sulphuric acid, would make the alloy pasty, and secure it from the action of the acid; and there would happen, besides, jumpings and startings, which might break the vessels. The alloy, purified by nitre, is granulated, and 12 kilogrammes = 26½ lbs. or 15 kilogrammes = 33 lbs. avoirdupois, are introduced into a platinum retort, and three times their weight of concentrated sulphuric acid, at 65^0 = 1.74 sp. gr. or 66^0 = 1.76 sp. gr., poured

upon them, and heated gradually to ebullition. At the end of three or four hours, the fire is drawn, and after one hour the liquid containing the copper and silver in solution is withdrawn, and the residual gold washed; but as it is not yet perfectly pure, it is treated a second time with sulphuric, and a third and last time by the same acid, to which a little nitric acid has been added. In a factory there are generally several alembics ranged together; a single fire is sufficient to heat three, the fumes passing into the same chimney; above each alembic is a head, whose neck communicates with a tube, leading to a channel lined with lead, the bottom of which is covered with water, and which is close to the furnace chimneys in which the nitre operation is executed; by these means the workmen are not incommoded by the sulphurous acid or by the vapours of sulphuric acid. The solutions of sulphate of silver are left to clear, and the muddy deposit which contains the gold is collected; these solutions are weakened with water until they mark at most $25^0 = 1.19$ sp. gr. of the areometer, and slips of copper, and then placed in them to precipitate the silver. It is known that the precipitation of this metal takes place but very slowly in very concentrated solutions, and the solutions of sulphate of copper, which mark more than $25^0 = 1.19$ sp. gr., exercise a sensible oxidating action on the silver.

The solutions of sulphate of copper are evaporated in leaden boilers, and crystallized, the salt purified by another crystallization. The acid mother waters are evaporated to $55^0 = 1.56$ sp. gr., and a little anhydrous sulphate of copper precipitates, and scarcely anything

remains in solution, and it is used for acting on the alloys as sulphuric acid.

The boilers of lead in which the silver is precipitated, are covered with a deposit of that metal, which is removed from time to time, washed, dried, and fused with 3 or 4 parts to the 100 nitre, and it produces fine silver.

By this process, a thousandth or even less of gold can be separated from silver. When the assay contains a thousandth, the refiners in France have the copper and the gold, and return the fine silver. When the assay is above this standard, the refiners return the gold and silver separate, and take, as the price of parting, 4.5 to 5 francs per kilogramme=2.2 lbs. avoirdupois.

In order to economise the consumption of sulphuric acid, M. Lebel, proprietor of the factory of Belleville, proposed roasting the alloy reduced to large grains, about the size of nuts, in a reverberating furnace, and then treating it in boilers of lead with weak sulphuric acid. This process has been practiced for three years at Freyburg, to separate the copper contained in the silver proceeding from the amalgamation, and succeeded very well, but required three roastings, and three successive treatments with acid, to produce a silver of .960. Nevertheless, as this silver contained besides copper, a small quantity of other metals, which injured its ductility, another mode of separation was followed.*

Dry parting.—The operation which is termed parting

* See Annales des Mines, vol. III. p. 15, n. s.

by the dry way, is never employed but in the treatment of very poor alloys of gold, in order to concentrate it into a less proportion of silver, and in order *pour donner la fausse*; that is to say, to separate the copper from the surfaces of bodies manufactured of alloys of that metal and gold, so that they may have the appearance of pure gold. This parting is made by cementation with chlorinised and oxidated bodies, and is named the *concentrated parting*, or by means of sulphur; it is then named the *dry parting*. The concentrated parting is scarcely practicable but *pour donner la fausse*. The object to be refined on the surface, or the alloy to be parted, is placed in a crucible; after having reduced it to thin plates, enveloping and stratifying it with a cement, of which the composition varies very much; some employ a mixture of equal parts of sulphate of iron and sea-salt; others a mixture of 2 parts of sea-salt; 2 parts of sulphate of iron, and 1 part of sal ammoniac; others, again, mix nitre and sulphate of iron in place of sea-salt; others, again, mix sulphate of iron, nitre, sea-salt, or sal ammoniac together. In all cases there is added to the mixture three or four times its weight of burnt clay, or of finely powdered brick, and the whole made into a thick paste. It is heated gradually, until of a dull red heat, and kept at that heat for twenty-four hours, great attention being paid to avoid the fusion or softening of the alloy. It is allowed to cool completely, and the alloy is removed; if not thought sufficiently rich, it is cemented a second time. When the cement contains a chloride, it is by the chlorine that the silver is acted upon; and when it contains oxidating matters, it is by the action of the nitric acid which

the nitre furnishes, that the silver is separated; and such is the action of this acid, in this case, that it will refine completely the surface of alloys which it will not touch in the *wet way*. The cement of nitre and sulphate of iron is also an excellent one for giving the *fausse*. If by this method it is wished to separate all the silver from the gold, it is necessary, after one cementation, to re-fuse the alloy, and laminate and cement as often as required, because the action of the oxidating mixture extends but to a certain depth, which is very small. The silver can be extracted from the scoriæ by fusing them with a reducing agent, mixed with litharge and potash.

Dry parting.—In the time of Schlutter, alloys of gold and silver were separated by *dry parting*, instead of submitting them to the *wet parting*, by means of nitric acid, when they contained $\frac{1}{4}$th of gold, more or less, and by this process could be extracted $\frac{1}{200}$th of gold contained in silver, so that no gold should be lost; that is to say, in order that it should not pass with the sulphuret of silver, it is necessary to limit the richness of the alloy to about 0.12 of gold.

In order to perform this operation, the alloy is fused and granulated very finely, by throwing it into a tub full of water, to which a quick rotatory motion is given by a broom. The granulated mass is mixed with a 0.12 to a 0.15 of flour of sulphur while still wet, and when it adheres to the surface, it is gradually heated to a dull red in a covered crucible. When it is thought that a sulphuret is for the greater part formed, the heat is increased until it enters into complete fusion, and the fused matter is poured into a conical iron mould,

slightly greased and heated. The mould is then reversed, and the matter examined. It sometimes happens that it is homogeneous, and then it is well sulphurized, and it is sometimes composed of a metallic alloy and a sulphuretted mass, called *plachnall;* in this second case, the alloy is of a convenient richness, or it is very rich, or on the contrary, it is not rich enough. When there is no button, the plachnall is re-fused, with the addition of a certain quantity of granulated alloy which has been kept in reserve, or with a small quantity of iron filings (0.04 to 0.05), and all the gold alloyed with a certain quantity of silver is thus separated. When the fusion with sulphur gives a button of suitable richness, the operation is complete. When this button is very rich, gold remains in the plachnall, which is to be re-fused with the granulated alloy or with iron. Lastly, when the button is too poor, it is treated a second time with sulphur. In order to purify the enriched alloy, it is heated strongly on a cupel, so as to decompose the small quantity of sulphuret with which it might be mixed, then to add 0.12 to 0.15 of lead, and cupel; lastly to extract the silver from the plachnall by fusing it with iron if it is very impure, or by roasting it, if it is nearly pure.

Standard of the alloys of gold.—The real standard of the alloys of gold is expressed in fractions of unity as is the case in alloys of silver. We suppose 24 carats in unity, and 32 32nds in the carat; the unity contains then 768 32nds. After these data the following Table has been formed, which expresses the relation of 32nds and carats to decimal fractions of the unity.

32nds.	Decimals.
1	0.001302
2	0.002604
3	0.003906
4	0.005208
5	0.006510
6	0.007912
7	0.009115
8	0.010415
9	0.011718
10	0.013021
11	0.014323
12	0.015625
13	0.016927
14	0.018230
15	0.019531
16	0.020833
17	0.022135
18	0.023436
19	0.024740
20	0.026042
21	0.027343
22	0.028644
23	0.029948
24	0.031250
25	0.032552
26	0.033854
27	0.035156
28	0.036460
29	0.037760
30	0.039062
31	0.040364
32	0.041667

Carats.	Decimals.
1	0.041667
2	0.083334
3	0.125001
4	0.166667
5	0.208333
6	0.250000
7	0.291666
8	0.333333
9	0.374999
10	0.416667
11	0.458630
12	0.500000
13	0.541667
14	0.583333
15	0.624555
16	0.666667
17	0.707333
18	0.750000
19	0.791666
20	0.833333
21	0.874999
22	0.916666
23	0.958333
24	1.000000

CHAPTER XVII.

ASSAY AND ANALYSIS OF FUEL.

THE substances generally employed as fuel, are wood, wood-charcoal, turf, turf-charcoal, coal, and its coke.

The essential elements of combustible matters are carbon, oxygen, and hydrogen; nitrogen being present sometimes, but only in small proportions. These constitute the organic part; various salts and silica constitute the inorganic part, or ash.

Gaseous products.—In order to estimate the amount of gas given off from any particular sample of coal, proceed in the following manner. Place a given weight, say 200 grs., of the coal in a red hot iron tube, closed at one end, to the other end of which, adapt by means of a cork, a glass or other tube, which must be conducted into an inverted jar, full of water, and standing in the pneumatic trough. Continue the red heat until no more gas is given off, then ascertain its quantity in cubic inches with due correction for temperature and pressure.

Amount of Coke.—The residue of the last operation is the amount of coke which that particular sample of coal produces; and its weight divided by two, gives the per centage of coke.

Amount of Ash.—Fully ignite about 50 grs. of coal in a platinum capsule, until nothing but ash is left. Its amount may then be ascertained by weighing.

Amount of Sulphur.—This is a most important operation in the assay; as a coal containing sulphur cannot be employed for particular purposes, and, indeed, those which contain much sulphur ought only to be used for the commonest purposes. This assay is most important to steam-boat and other companies who consume fuel under steam-boilers; and the coal they purchase should always be subjected to this particular test, as sulphur has a most corroding and destroying action on iron and copper. Where sulphur coals are continually burnt under boilers, the metal of the latter becomes so speedily deteriorated, that the boiler is rapidly rendered useless.

Process.—1 part of the coal, finely pulverized, is mixed with 7 or 8 parts of nitrate of potash and 16 parts of common salt, and 4 parts of carbonate of potash, all of which saline matters must be perfectly pure; the mixture is then placed in a platinum crucible and gently heated. At a certain temperature, the whole ignites and burns quietly; the operation is finished when the mass is white. It must, when cold, be dissolved in water, the solution slightly acidulated by means of hydrochloric acid, and chloride of barium added to it as long as a white precipitate forms. This precipitate is sulphate of baryta, which must be collected on a filter, washed, dried, ignited, and weighed; every 116 parts of it indicate 16 of sulphur.

Heating power of Coal.—Equal weights of different combustibles give off, when burned, very unequal

quantities of heat. Their heating, or calorific power, is equal to these quantities. Sometimes these quantities are ascertained by the quantity of water evaporated, and sometimes by the amount of ice melted, and at others by the quantity of water raised 1^0 of the thermometer.

According to Berthier, however, the most convenient method as ascertaining the comparative calorific powers of any combustible matters, is by means of litharge. He says: "It has been proved by the experiments of many philosophers, that the quantities of heat emitted by combustible substances, are exactly proportioned to the amounts of oxygen required for their complete combustion. From whence, after the elementary constitution of any combustible is known, its calorific power is easily determined by calculation. For instance, it is only necessary to ascertain the quantity of oxygen absorbed in the conversion of all its carbon into carbonic acid, and all its hydrogen into water, and compare that quantity with that which is consumed in burning a fuel whose calorific power is well ascertained: such a substance is charcoal."

By adopting the principle just pointed out, it may be conceived, that without knowing the composition of a fuel, its heating power may be ascertained by determining the amount of oxygen it absorbs in burning. This can be done in a very simple and expeditious manner, if not exactly, at least with sufficient exactitude to afford very useful results in practice. It is as follows:—Many metallic oxides are reduced with such facility, that when heated with a combustible body, the latter burns completely, without any of its elements

escaping the action of the oxygen of the oxide, if the operation be performed suitably. The composition of the oxide being well known, if the weight of the part reduced to the metallic state be taken, the quantity of oxygen employed in the combustion can be ascertained. In order to collect the metal, and separate it from the non-reduced mass, it must be fusible as well as its oxide. Litharge fulfils these conditions, and experiment has proved that it completely burns the greater part of all ordinary fuels; the only exceptions are some very bituminous matters, which containing a large proportion of volatile elements, allow a portion to escape before the temperature is sufficiently high to allow the reduction to take place. The experiment is made as follows:—

One part, say 10 grs., of the finely powdered or otherwise divided fuel, is mixed with about 400 grs. of litharge. The mixture is carefully placed in an earthen crucible, and covered with 200 grs. more litharge. The crucible is then placed in the fire, and gradually heated. When the fusion is perfect, the heat is urged for about ten minutes, in order that all the lead may collect into a single button. The crucible is then taken from the fire, cooled, broken, and the button of lead weighed. Sometimes the button of lead is livid, leafy, and only slightly ductile; in which case, it has absorbed a little litharge. This I find, can be partially prevented by fusing slowly, and adding a little borax.

Two assays, at least, ought to be made; and those results which differ more than a grain or two, ought not to be relied on. The purer the litharge, the better the result; it ought to contain as little minium as pos-

sible. It is an excellent plan to mix up the litharge of commerce with one or two thousandths of its weight of charcoal, and fuse the whole in a pot: when cold, pulverize the altered litharge, which will now be deprived of minium.

Pure carbon produces with pure litharge thirty-four times its weight of lead, and hydrogen 103.7 times its weight of lead, that is to say, a little more than three times as much as carbon. We can, therefore, from these data, find the equivalent of any fuel, either in carbon or hydrogen.

When a fuel contains volatile matters, the quantity can be ascertained, as before pointed out, by ignition in a close tube or crucible. If, further, we ascertain the proportion of lead it gives with litharge, it is easy to calculate the equivalent in carbon of the volatile matters, and, in consequence, to ascertain its calorific value.

Supposing that a substance gives by distillation C of coke or carbon, having deducted the weight of the ash and V of volatile substances, and that it produces P of lead with litharge. The quantity C of carbon would give $34 \times C$ of lead; the quantity of volatile matter would give but $P - 34 \times C$; it would be equivalent then to $\frac{P - 34 \times C}{34}$ of carbon; from whence it follows, that the quantity of heat developed by the charcoal, the volatile matter, and the unaltered combustible, will be to each other as the numbers $34 \times C$, $P - 34 \times C$ and P, which represents the quantities of lead; or as the numbers $C, \frac{P - 34 \times C}{34}, \frac{P}{34}$ which represents the quantities of carbon.

Dr. Ure* says, speaking of the above method of assay: "On subjecting this theory to the touchstone of experiment, I have found it to be entirely fallacious. Having mixed, very intimately, 10 grains of recently calcined charcoal with 1000 parts of litharge, both in fine powder, I placed the mixture in a crucible, which was so carefully covered, as to be protected from all fuliginous fumes, and exposed it to distinct ignition.

"No less than 603 grains of lead were obtained; whereas, by Berthier's rule, only 340 or 346.6 were possible. On igniting a mixture of 10 grains of pulverized anthracite, from Merthyr Tydfill, with 500 grs. of pure litharge, previously fused and pulverized, I obtained 380 grains of metallic lead. In a second experiment, with the same anthracite and the same litharge, I obtained 450 grains of lead; and in a third, only 350 grains. It is, therefore, obvious that this method of Berthier's is altogether nugatory for ascertaining the quantity of carbon in coals, and is worse than useless in judging of the calorific qualities of different kinds of fuel."

This discrepancy in the results obtained by Dr. Ure is very perplexing, and does not at all accord with Berthier's experience, as shown by his experiments, or by my own on the subject. I have never had a difference of more than 50 grains, and in general, only 2 or 3, which latter result is satisfactory. The only precaution I have found necessary, is to heat very gradually until the mixture be fully fused, and then to increase the

* Supplement to the Dictionary of Arts, Mines, and Manufactures.

fire to bright redness for a few minutes. I shall, however, make a more extended series of experiments on this subject, with the view of perfecting the process, as it is at once easy, expeditious, and would be exceedingly valuable, could it be rendered certain.*

The following is the method of ascertaining the calorific power of fuel employed by Dr. Ure, and described in his Supplement.

"The following calorimeter, founded upon the same principle as that of Count Rumford, but with certain improvements, may be considered as an equally correct instrument for measuring heat with any of the preceding (Lavoisier, Meyers, and others), but one of much more general application, since it can determine the quantity of heat disengaged in combustion, as well as the latent heat of steam and other vapours.

"It consists of a large copper bath, capable of holding 100 gallons of water. It is traversed four times backwards and forwards, in four different levels, by a

* Since writing the above, I have made many experiments on this subject, and succeed most perfectly in estimating the value of a fuel. With the litharge of commerce, which contains much minium, the process is never exact; I have obtained results differing as much as 40 or 50 grains, when I have not purified the litharge employed, and to purify it completely is a troublesome process. This difficulty I have completely obviated, however, b substituting for litharge the white lead of commerce, using for each 10 grains of fuel, 700 grains of white lead, which are well mixed with it, and 300 grains of pure white lead to cover the mixture. When the whole is heated, the carbonate of lead decomposes, forming pure oxide of lead, which is then reduced, as in the former case. By this process, the results correspond to 1 grain in the quantity of lead produced from a given sample of fuel.

zig-zag horizontal flue, or flat pipe, 9 inches broad, and 1 deep, ending below in a round pipe, which passes through the bottom of the bath, and receives there into it the top of a small black-lead furnace, the innermost crucible of which contains the fuel. It is surrounded, at the distance of an inch, by a second crucible, which is enclosed, at the same time, by the sides of the outermost furnace, the strata of stagnant air between the crucibles serving to prevent the heat being dissipated into the atmosphere by the body of the furnace. A pipe from a double pair of bellows enters the ash-pit of the furnace at one side, and supplies a steady but gentle heat to carry on the combustion kindled at first by half an ounce of burning charcoal. So completely is the heat which is disengaged by the burning fuel absorbed by the water in the bath, that the air discharged at the top pipe is generally the same temperature as the atmosphere. The vessel is made of copper, weighing 2 lbs. per square foot; it is $5\frac{1}{2}$ feet long, $1\frac{1}{2}$ wide, 2 deep, with a bottom $5\frac{1}{2}$ feet long, and $1\frac{3}{4}$ broad upon an average. Including the zig-zag tin-plate flue, and a rim of wrought iron, it weighs altogether 85 lbs. Since the specific heat of copper is to that of water as 94 to 1000, the specific heat of the vessel is equal to that of 8 lbs. of water; for which, therefore, the exact correction is made by leaving 8 lbs. of water out of the 600 or 1000 lbs. used in each experiment.

"In the experiments made with former calorimeters of this kind, the combustion was maintained by the current or draught of a chimney open at bottom, which carried off at the top orifice of the flue a variable quantity of heat, very difficult to estimate.

"The heating power of the fuel is measured by the number of degrees of temperature which the combustion of 1 lb. of it raises 600 or 1000 lbs. of water in the bath, the copper substance of the vessel being taken into account. One pound of dry wood charcoal, by its combustion, causes 6000 lbs. of water to become 20^0 hotter. For the sake of brevity, we shall call this calorific energy 12,000 unities. In like circumstance, 1 lb. of Llangennock coal will yield by combustion 11,500 unities of caloric."

This form of calorimeter of Dr. Ure's seems to possess many advantages over Laplace's and others; and is, no doubt, very convenient in use, although rather bulky.

CHAPTER XVIII.

ASSAY TABLE,

SHOWING THE AMOUNT OF GOLD OR SILVER, IN OUNCES, PENNYWEIGHTS, AND GRAINS, CONTAINED IN A TON OF ORE, &c., FROM THE WEIGHT OF METAL OBTAINED IN AN ASSAY OF 200 GRAINS OF MINERAL.

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.001	0	3	6	.019	3	2	1
.002	0	6	12	.020	3	5	8
.003	0	9	19	.021	3	8	14
.004	0	13	1	.022	3	11	20
.005	0	16	8	.023	3	15	3
.006	0	19	14	.024	3	18	9
.007	1	2	20	.025	4	1	16
.008	1	6	3	.026	4	4	22
.009	1	9	9	.027	4	8	4
.010	1	12	6	.028	4	11	11
.011	1	15	22	.029	4	14	17
.012	1	19	4	.030	4	18	0
.013	2	2	11	.031	5	1	6
.014	2	5	17	.032	5	4	12
.015	2	9	0	.033	5	7	19
.016	2	12	6	.034	5	11	1
.017	2	15	12	.035	5	14	8
.018	2	18	19	.036	5	17	14

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.037	6	0	20
.038	6	4	3
.039	6	7	9
.040	6	10	16
.041	6	13	22
.042	6	17	4
.043	7	0	11
.044	7	3	17
.045	7	7	0
.046	7	10	6
.047	7	13	12
.048	7	16	19
.049	8	0	1
.050	8	3	8
.051	8	6	14
.052	8	9	20
.053	8	13	3
.054	8	16	9
.055	8	19	16
.056	9	2	22
.057	9	6	4
.058	9	9	11
.059	9	12	17
.060	9	16	0
.061	9	19	6
.062	10	2	12
.063	10	5	19
.064	10	9	1
.065	10	12	8
.066	10	15	14
.067	10	18	20
.068	11	2	3
.069	11	5	9
.070	11	8	16

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.071	11	11	22
.072	11	15	4
.073	11	18	11
.074	12	1	17
.075	12	5	0
.076	12	8	6
.077	12	11	12
.078	12	14	19
.079	12	18	1
.080	13	1	8
.081	13	4	14
.082	13	7	20
.083	13	11	3
.084	13	14	9
.085	13	17	16
.086	14	0	22
.087	14	4	4
.088	14	7	11
.089	14	10	17
.090	14	14	0
.091	14	17	6
.092	15	0	12
.093	15	3	19
.094	15	7	1
.095	15	10	8
.096	15	13	14
.097	15	16	20
.098	16	0	3
.099	16	3	9
.100	16	6	16
.101	16	9	22
.102	16	13	4
.103	16	16	11
.104	16	19	17

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.105	17	3	0	.139	22	14	1
.106	17	6	6	.140	22	17	8
.107	17	9	12	.141	23	0	14
.108	17	12	19	.142	23	3	20
.109	17	16	1	.143	23	7	3
.110	17	19	8	.144	23	10	9
.111	18	2	14	.145	23	13	16
.112	18	5	20	.146	23	16	22
.113	18	9	3	.147	24	0	4
.114	18	12	9	.148	24	3	11
.115	18	15	16	.149	24	6	17
.116	18	18	22	.150	24	10	0
.117	19	2	4	.151	24	13	6
.118	19	5	11	.152	24	16	12
.119	19	8	17	.153	24	19	19
.120	19	12	0	.154	25	3	1
.121	19	15	6	.155	25	6	8
.122	19	18	12	.156	25	9	14
.123	20	1	19	.157	25	12	20
.124	20	5	1	.158	25	16	3
.125	20	8	8	.159	25	19	9
.126	20	11	14	.160	26	2	16
.127	20	14	20	.161	26	5	22
.128	20	18	3	.162	26	9	4
.129	21	1	9	.163	26	12	11
.130	21	4	16	.164	26	15	17
.131	21	7	22	.165	26	19	0
.132	21	11	4	.166	27	2	6
.133	21	14	11	.167	27	5	12
.134	21	17	17	.168	27	8	19
.135	22	1	0	.169	27	12	1
.136	22	4	6	.170	27	15	8
.137	22	7	12	.171	27	18	14
.138	22	10	19	.172	28	1	20

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.173	28	5	3
.174	28	8	9
.175	28	11	16
.176	28	14	22
.177	28	18	4
.178	29	1	11
.179	29	4	17
.180	29	8	0
.181	29	11	6
.182	29	14	12
.183	29	17	19
.184	30	1	1
.185	30	4	8
.186	30	7	14
.187	30	10	20
.188	30	14	3
.189	30	17	9
.190	31	0	16
.191	31	3	22
.192	31	7	4
.193	31	10	11
.194	31	13	17
.195	31	17	0
.196	32	0	6
.197	32	3	12
.198	32	6	19
.199	32	10	1
.200	32	13	8
.201	32	16	14
.202	32	19	20
.203	33	3	3
.204	33	6	9
.205	33	9	16
.206	33	12	22

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.207	33	16	4
.208	33	19	11
.209	34	2	17
.210	34	6	0
.211	34	9	6
.212	34	12	12
.213	34	15	19
.214	34	19	1
.215	35	2	8
.216	35	5	14
.217	35	8	20
.218	35	12	3
.219	35	15	9
.220	35	18	16
.221	36	1	22
.222	36	5	4
.223	36	8	11
.224	36	11	17
.225	36	15	0
.226	36	18	6
.227	37	1	12
.228	37	4	19
.229	37	8	1
.230	37	11	8
.231	37	14	14
.232	37	17	20
.233	38	1	3
.234	38	4	9
.235	38	7	16
.236	38	10	22
.237	38	14	4
.238	38	17	11
.239	39	0	17
.240	39	4	0

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.241	39	7	6	.275	44	18	8
.242	39	10	12	.276	45	1	14
.243	39	13	18	.277	45	4	20
.244	39	17	1	.278	45	8	3
.245	40	0	8	.279	45	11	9
.246	40	3	14	.280	45	14	16
.247	40	6	20	.281	45	17	22
.248	40	10	3	.282	46	1	4
.249	40	13	9	.283	46	4	11
.250	40	16	16	.284	46	7	17
.251	40	19	22	.285	46	11	0
.252	41	3	4	.286	46	14	6
.253	41	6	11	.287	46	17	12
.254	41	9	17	.288	47	0	19
.255	41	13	0	.289	47	4	1
.256	41	16	6	.290	47	7	8
.257	41	19	12	.291	47	10	14
.258	42	2	19	.292	47	13	20
.259	42	6	1	.293	47	17	3
.260	42	9	8	.294	48	0	9
.261	42	12	14	.295	48	3	16
.262	42	15	20	.296	48	6	22
.263	42	19	3	.297	48	10	4
.264	43	2	9	.298	48	13	11
.265	43	5	16	.299	48	16	17
.266	43	8	22	.300	49	0	0
.267	43	12	4	.301	49	3	6
.268	43	15	11	.302	49	6	12
.269	43	18	17	.303	49	9	19
.270	44	2	0	.304	49	13	1
.271	44	5	6	.305	49	16	8
.272	44	8	12	.306	49	19	14
.273	44	11	19	.307	50	2	20
.274	44	15	1	.308	50	6	3

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.309	50	9	9	.343	56	0	11
.310	50	12	16	.344	56	3	17
.311	50	15	22	.345	56	7	0
.312	50	19	4	.346	56	10	6
.313	51	2	11	.347	56	13	12
.314	51	5	17	.348	56	16	19
.315	51	9	0	.349	57	0	1
.316	51	12	6	.350	57	3	8
.317	51	15	12	.351	57	6	14
.318	51	18	19	.352	57	9	20
.319	52	2	1	.353	57	13	3
.320	52	5	8	.354	57	16	9
.321	52	8	14	.355	57	19	16
.322	52	11	20	.356	58	2	22
.323	52	15	3	.357	58	6	4
.324	52	18	9	.358	58	9	11
.325	53	1	16	.359	58	12	17
.326	53	4	22	.360	58	16	0
.327	53	8	4	.361	58	19	6
.328	53	11	11	.362	59	2	12
.329	53	14	17	.363	59	5	19
.330	53	18	0	.364	59	9	1
.331	54	1	6	.365	59	12	8
.332	54	4	12	.366	59	15	14
.333	54	7	19	.367	59	18	20
.334	54	11	1	.368	60	2	3
.335	54	14	8	.369	60	5	9
.336	54	17	14	.370	60	8	16
.337	55	0	20	.371	60	11	22
.338	55	4	3	.372	60	15	4
.339	55	7	9	.373	60	18	11
.340	55	10	16	.374	61	1	17
.341	55	13	22	.375	61	5	0
.342	55	17	4	.376	61	8	6

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.377	61	11	12	.411	67	2	14
.378	61	14	19	.412	67	5	20
.379	61	18	1	.413	67	9	3
.380	62	1	8	.414	67	12	9
.381	62	4	14	.415	67	15	16
.382	62	7	20	.416	67	18	22
.383	62	11	3	.417	68	2	4
.384	62	14	9	.418	68	5	11
.385	62	17	16	.419	68	8	17
.386	63	0	22	.420	68	12	0
.387	63	4	4	.421	68	15	6
.388	63	7	11	.422	68	18	12
.389	63	10	17	.423	69	1	19
.390	63	14	0	.424	69	5	1
.391	63	17	6	.425	69	8	8
.392	64	0	12	.426	69	11	14
.393	64	3	19	.427	69	14	20
.394	64	7	1	.428	69	18	3
.395	64	10	8	.429	70	1	9
.396	64	13	14	.430	70	4	16
.397	64	16	20	.431	70	7	22
.398	65	0	3	.432	70	11	4
.399	65	3	9	.433	70	14	11
.400	65	6	16	.434	70	17	17
.401	65	9	22	.435	71	1	0
.402	65	13	4	.436	71	4	6
.403	65	16	11	.437	71	7	12
.404	65	19	17	.438	71	10	19
.405	66	3	0	.439	71	14	1
.406	66	6	6	.440	71	17	8
.407	66	9	12	.441	72	0	14
.408	66	12	19	.442	72	3	20
.409	66	16	1	.443	72	7	3
.410	66	19	8	.444	72	10	9

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.445	72	13	16
.446	72	16	22
.447	73	0	4
.448	73	3	11
.449	73	6	17
.450	73	10	0
.451	73	13	6
.452	73	16	12
.453	73	19	19
.454	74	3	1
.455	74	6	8
.456	74	9	14
.457	74	12	20
.458	74	16	3
.459	74	19	9
.460	75	2	16
.461	75	5	22
.462	75	9	4
.463	75	12	11
.464	75	15	17
.465	75	19	0
.466	76	2	6
.467	76	5	12
.468	76	8	19
.469	76	12	1
.470	76	15	8
.471	76	18	14
.472	77	1	20
.473	77	5	3
.474	77	8	9
.475	77	11	16
.476	77	14	22
.477	77	18	4
.478	78	1	11

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.479	78	4	17
.480	78	8	0
.481	78	11	6
.482	78	14	12
.483	78	17	19
.484	79	1	1
.485	79	4	8
.486	79	7	14
.487	79	10	20
.488	79	14	3
.489	79	17	9
.490	80	0	16
.491	80	3	22
.492	80	7	4
.493	80	10	11
.494	80	13	17
.495	80	17	0
.496	81	0	6
.497	81	3	12
.498	81	6	19
.499	81	10	1
.500	81	13	8
.501	81	16	14
.502	81	19	20
.503	82	3	3
.504	82	6	9
.505	82	9	16
.506	82	12	22
.507	82	16	4
.508	82	19	11
.509	83	2	17
.510	83	6	0
.511	83	9	6
512	83	12	12

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.513	83	15	19	.547	89	6	20
.514	83	19	1	.548	89	10	3
.515	84	2	8	.549	89	13	9
.516	84	5	14	.550	89	16	16
.517	84	8	20	.551	89	19	22
.518	84	12	3	.552	90	3	4
.519	84	15	9	.553	90	6	11
.520	84	18	16	.554	90	9	17
.521	85	1	22	.555	90	13	0
.522	85	5	4	.556	90	16	6
.523	85	8	11	.557	90	19	12
.524	85	11	17	.558	91	2	19
.525	85	15	0	.559	91	6	1
.526	85	18	6	.560	91	9	8
.527	86	1	12	.561	91	12	14
.528	86	4	19	.562	91	15	20
.529	86	8	1	.563	91	19	3
.530	86	11	8	.564	92	2	9
.531	86	14	14	.565	92	5	16
.532	86	17	20	.566	92	8	22
.533	87	1	3	.567	92	12	4
.534	87	4	9	.568	92	15	11
.535	87	7	16	.569	92	18	17
.536	87	10	22	.570	93	2	0
.537	87	14	4	.571	93	5	6
.538	87	17	11	.572	93	8	12
.539	88	0	17	.573	93	11	19
.540	88	4	0	.574	93	15	1
.541	88	7	6	.575	93	18	8
.542	88	10	12	.576	94	1	14
.543	88	13	19	.577	94	4	20
.544	88	17	1	.578	94	8	3
.545	89	0	8	.579	94	11	9
.546	89	3	14	.580	94	14	16

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.581	94	17	22
.582	95	1	4
.583	95	4	11
.584	95	7	17
.585	95	11	0
.586	95	14	6
.587	95	17	12
.588	96	0	19
.589	96	4	1
.590	96	7	8
.591	96	10	14
.592	96	13	20
.593	96	17	3
.594	97	0	9
.595	97	3	16
.596	97	6	22
.597	97	10	4
.598	97	13	11
.599	97	16	17
.600	98	0	0
.601	98	3	6
.602	98	6	12
.603	98	9	19
.604	98	13	1
.605	98	16	8
.606	98	19	14
.607	99	2	20
.608	99	6	3
.609	99	9	9
.610	99	12	16
.611	99	15	22
.612	99	19	4
.613	100	2	11
.614	100	5	17

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.615	100	9	0
.616	100	12	6
.617	100	15	12
.618	100	18	19
.619	101	2	1
.620	101	5	8
.621	101	8	14
.622	101	11	20
.623	101	15	3
.624	101	18	9
.625	102	1	16
.626	102	4	22
.627	102	8	4
.628	102	11	11
.629	102	14	17
.630	102	18	0
.631	103	1	6
.632	103	4	12
.633	103	7	19
.634	103	11	1
.635	103	14	8
.636	103	17	14
.637	104	0	20
.638	104	4	3
.639	104	7	9
.640	104	10	16
.641	104	13	22
.642	104	17	4
.643	105	0	11
.644	105	3	17
.545	105	7	0
.546	105	10	6
.647	105	13	12
.648	105	16	19

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.649	106	0	1	.683	111	11	3
.650	106	3	8	.684	111	14	9
.651	106	6	14	.685	111	17	6
.652	106	9	20	.686	112	0	22
.653	106	13	3	.687	112	4	4
.654	106	16	9	.688	112	7	11
.655	106	19	16	.689	112	10	17
.656	107	2	22	.690	112	14	0
.657	107	6	4	.691	112	17	6
.658	107	9	11	.692	113	0	12
.659	107	12	17	.693	113	3	19
.660	107	16	0	.694	113	7	1
.661	107	19	6	.695	113	10	8
.662	108	2	12	.696	113	13	14
.663	108	5	19	.697	113	16	20
.664	108	9	1	.698	114	0	3
.665	108	12	8	.699	114	3	9
.665	108	15	14	.700	114	6	16
.667	108	18	20	.701	114	9	22
.668	109	2	3	.702	114	13	4
.669	109	5	9	.703	114	16	12
.670	109	8	16	.704	114	19	17
.671	109	11	22	.705	115	3	0
.672	109	15	4	.706	115	6	6
.673	109	18	11	.707	115	9	12
.674	110	1	17	.708	115	12	19
.675	110	5	0	.909	115	16	1
.676	110	8	6	.710	115	19	8
.677	110	11	12	.711	116	2	14
.678	110	14	19	.712	116	5	20
.679	110	18	1	.713	116	9	3
.680	111	1	8	.714	116	12	9
.681	111	4	14	.715	116	15	16
.682	111	7	20	.716	116	18	22

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.717	117	2	4
.718	117	5	11
.719	117	8	17
.720	117	12	0
.721	117	15	6
.722	117	18	12
.723	118	1	19
.724	118	5	1
.725	118	8	8
.726	118	11	14
.727	118	14	20
.728	118	18	3
.729	119	1	9
.730	119	4	16
.731	119	7	22
.732	119	11	4
.733	119	14	11
.734	119	17	17
.735	120	1	0
.736	120	4	6
.737	120	7	12
.738	120	10	19
.739	120	14	1
.740	120	17	2
.741	121	0	14
.742	121	3	20
.743	121	7	3
.744	121	10	9
.745	121	13	6
.746	121	16	22
.747	122	0	4
.748	122	3	11
.749	122	6	17
.750	122	10	0

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.751	122	13	16
.752	122	16	12
.753	122	19	19
.754	123	3	1
.755	123	6	8
.756	123	9	14
.757	123	12	20
.758	123	16	3
.759	123	19	9
.760	124	2	16
.761	124	5	22
.762	124	9	4
.763	124	12	11
.764	124	15	17
.765	124	19	0
.766	125	2	6
.767	125	5	12
.768	125	8	19
.769	125	12	1
.770	125	15	8
.771	125	18	14
.772	126	1	20
.773	126	5	3
.774	126	8	9
.775	126	11	16
.776	126	14	22
.777	126	18	4
.778	127	1	11
.779	127	4	17
.780	127	8	0
.781	127	11	6
.782	127	14	12
.783	127	17	19
.784	128	1	1

If 200 grains of ore give: FINE METAL.	One ton of ore will yield: FINE METAL.		
gr.	oz.	dwts.	grs.
.785	128	4	8
.786	128	7	14
.787	128	10	20
.788	128	14	3
.789	128	17	9
.790	129	0	16
.791	129	3	22
.792	129	7	4
.793	129	10	11
.794	129	13	17
.795	129	17	0
.796	130	0	6
.797	130	3	12
.798	130	6	19
.799	130	10	1
.800	130	13	8
.801	130	16	14
.802	130	19	20
.803	131	3	3
.804	131	6	9
.805	131	9	16
.806	131	12	22
.807	131	16	4
.808	131	19	11
.809	132	2	17
.810	132	6	0
.811	132	9	6
.812	132	12	12
.813	132	15	19
.814	132	19	1
.815	133	2	8
.816	133	5	14
.817	133	8	20
.818	133	12	3

If 200 grains of ore give: FINE METAL.	One ton of ore will yield: FINE METAL.		
gr.	oz.	dwts.	grs.
.819	133	15	9
.820	133	18	16
.821	134	1	22
.822	134	5	4
.823	134	8	11
.824	134	11	17
.825	134	15	0
.826	134	18	6
.827	135	1	12
.828	135	4	19
.829	135	8	1
.830	135	11	8
.831	135	14	14
.832	135	17	20
.833	136	1	3
.834	136	4	9
.835	136	7	16
.836	136	10	22
.837	136	14	4
.838	136	17	11
.839	137	0	17
.840	137	4	0
.841	137	7	6
.842	137	10	12
.843	137	13	19
.844	137	17	1
.845	138	0	8
.846	138	3	14
.847	138	6	20
.848	138	10	3
.849	138	13	19
.850	138	16	16
.851	138	19	22
.852	139	3	4

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.853	139	6	11
.854	139	9	17
.855	139	13	0
.856	139	16	6
.857	139	19	12
.858	140	2	19
.859	140	6	1
.860	140	9	8
.861	140	12	14
.862	140	15	20
.863	140	19	3
.864	141	2	9
.865	141	5	16
.866	141	8	22
.867	141	12	4
.868	141	15	11
.869	141	18	17
.870	142	2	0
.871	142	5	6
.872	142	8	12
.873	142	11	19
.874	142	15	1
.875	142	18	8
.876	143	1	14
.877	143	4	20
.878	143	8	3
.879	143	11	9
.880	143	14	16
.881	143	17	22
.882	144	1	4
.883	144	4	11
.884	144	7	17
.885	144	11	0
.886	144	14	6

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.887	144	17	12
.888	145	0	19
.889	145	4	1
.890	145	7	8
.891	145	10	14
.892	145	13	20
.893	145	17	3
.894	146	0	9
.895	146	3	16
.896	146	6	22
.897	146	10	4
.898	146	13	11
.899	146	16	17
.900	147	0	0
.901	147	3	6
.902	147	6	12
.903	147	9	19
.904	147	13	1
.905	147	16	8
.906	147	19	14
.907	148	2	2
.908	148	6	3
.909	148	9	9
.910	148	12	16
.911	148	15	21
.912	148	19	4
.913	149	2	11
.914	149	5	17
.915	149	9	0
.916	149	12	6
.917	149	15	12
.918	149	18	19
.919	150	2	1
.920	150	5	8

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.241	39	7	6	.275	44	18	8
.242	39	10	12	.276	45	1	14
.243	39	13	18	.277	45	4	20
.244	39	17	1	.278	45	8	3
.245	40	0	8	.279	45	11	9
.246	40	3	14	.280	45	14	16
.247	40	6	20	.281	45	17	22
.248	40	10	3	.282	46	1	4
.249	40	13	9	.283	46	4	11
.250	40	16	16	.284	46	7	17
.251	40	19	22	.285	46	11	0
.252	41	3	4	.286	46	14	6
.253	41	6	11	.287	46	17	12
.254	41	9	17	.288	47	0	19
.255	41	13	0	.289	47	4	1
.256	41	16	6	.290	47	7	8
.257	41	19	12	.291	47	10	14
.258	42	2	19	.292	47	13	20
.259	42	6	1	.293	47	17	3
.260	42	9	8	.294	48	0	9
.261	42	12	14	.295	48	3	16
.262	42	15	20	.296	48	6	22
.263	42	19	3	.297	48	10	4
.264	43	2	9	.298	48	13	11
.265	43	5	16	.299	48	16	17
.266	43	8	22	.300	49	0	0
.267	43	12	4	.301	49	3	6
.268	43	15	11	.302	49	6	12
.269	43	18	17	.303	49	9	19
.270	44	2	0	.304	49	13	1
.271	44	5	6	.305	49	16	8
.272	44	8	12	.306	49	19	14
.273	44	11	19	.307	50	2	20
.274	44	15	1	.308	50	6	3

If 200 grains of ore give: FINE METAL.	One ton of ore will yield: FINE METAL.			If 200 grains of ore give: FINE METAL.	One ton of ore will yield: FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.309	50	9	9	.343	56	0	11
.310	50	12	16	.344	56	3	17
.311	50	15	22	.345	56	7	0
.312	50	19	4	.346	56	10	6
.313	51	2	11	.347	56	13	12
.314	51	5	17	.348	56	16	19
.315	51	9	0	.349	57	0	1
.316	51	12	6	.350	57	3	8
.317	51	15	12	.351	57	6	14
.318	51	18	19	.352	57	9	20
.319	52	2	1	.353	57	13	3
.320	52	5	8	.354	57	16	9
.321	52	8	14	.355	57	19	16
.322	52	11	20	.356	58	2	22
.323	52	15	3	.357	58	6	4
.324	52	18	9	.358	58	9	11
.325	53	1	16	.359	58	12	17
.326	53	4	22	.360	58	16	0
.327	53	8	4	.361	58	19	6
.328	53	11	11	.362	59	2	12
.329	53	14	17	.363	59	5	19
.330	53	18	0	.364	59	9	1
.331	54	1	6	.365	59	12	8
.332	54	4	12	.366	59	15	14
.333	54	7	19	.367	59	18	20
.334	54	11	1	.368	60	2	3
.335	54	14	8	.369	60	5	9
.336	54	17	14	.370	60	8	16
.337	55	0	20	.371	60	11	22
.338	55	4	3	.372	60	15	4
.339	55	7	9	.373	60	18	11
.340	55	10	16	.374	61	1	17
.341	55	13	22	.375	61	5	0
.342	55	17	4	.376	61	8	6

If 200 grains of ore give: FINE METAL.	One ton of ore will yield: FINE METAL.		
gr.	oz.	dwts.	grs.
.377	61	11	12
.378	61	14	19
.379	61	18	1
.380	62	1	8
.381	62	4	14
.382	62	7	20
.383	62	11	3
.384	62	14	9
.385	62	17	16
.386	63	0	22
.387	63	4	4
.388	63	7	11
.389	63	10	17
.390	63	14	0
.391	63	17	6
.392	64	0	12
.393	64	3	19
.394	64	7	1
.395	64	10	8
.396	64	13	14
.397	64	16	20
.398	65	0	3
.399	65	3	9
.400	65	6	16
.401	65	9	22
.402	65	13	4
.403	65	16	11
.404	65	19	17
.405	66	3	0
.406	66	6	6
.407	66	9	12
.408	66	12	19
.409	66	16	1
.410	66	19	8

If 200 grains of ore give: FINE METAL.	One ton of ore will yield: FINE METAL.		
gr.	oz.	dwts.	grs.
.411	67	2	14
.412	67	5	20
.413	67	9	3
.414	67	12	9
.415	67	15	16
.416	67	18	22
.417	68	2	4
.418	68	5	11
.419	68	8	17
.420	68	12	0
.421	68	15	6
.422	68	18	12
.423	69	1	19
.424	69	5	1
.425	69	8	8
.426	69	11	14
.427	69	14	20
.428	69	18	3
.429	70	1	9
.430	70	4	16
.431	70	7	22
.432	70	11	4
.433	70	14	11
.434	70	17	17
.435	71	1	0
.436	71	4	6
.437	71	7	12
.438	71	10	19
.439	71	14	1
.440	71	17	8
.441	72	0	14
.442	72	3	20
.443	72	7	3
.444	72	10	9

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.445	72	13	16
.446	72	16	22
.447	73	0	4
.448	73	3	11
.449	73	6	17
.450	73	10	0
.451	73	13	6
.452	73	16	12
.453	73	19	19
.454	74	3	1
.455	74	6	8
.456	74	9	14
.457	74	12	20
.458	74	16	3
.459	74	19	9
.460	75	2	16
.461	75	5	22
.462	75	9	4
.463	75	12	11
.464	75	15	17
.465	75	19	0
.466	76	2	6
.467	76	5	12
.468	76	8	19
.469	76	12	1
.470	76	15	8
.471	76	18	14
.472	77	1	20
.473	77	5	3
.474	77	8	9
.475	77	11	16
.476	77	14	22
.477	77	18	4
.478	78	1	11

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.479	78	4	17
.480	78	8	0
.481	78	11	6
.482	78	14	12
.483	78	17	19
.484	79	1	1
.485	79	4	8
.486	79	7	14
.487	79	10	20
.488	79	14	3
.489	79	17	9
.490	80	0	16
.491	80	3	22
.492	80	7	4
.493	80	10	11
.494	80	13	17
.495	80	17	0
.496	81	0	6
.497	81	3	12
.498	81	6	19
.499	81	10	1
.500	81	13	8
.501	81	16	14
.502	81	19	20
.503	82	3	3
.504	82	6	9
.505	82	9	16
.506	82	12	22
.507	82	16	4
.508	82	19	11
.509	83	2	17
.510	83	6	0
.511	83	9	6
512	83	12	12

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.513	83	15	19	.547	89	6	20
.514	83	19	1	.548	89	10	3
.515	84	2	8	.549	89	13	9
.516	84	5	14	.550	89	16	16
.517	84	8	20	.551	89	19	22
.518	84	12	3	.552	90	3	4
.519	84	15	9	.553	90	6	11
.520	84	18	16	.554	90	9	17
.521	85	1	22	.555	90	13	0
.522	85	5	4	.556	90	16	6
.523	85	8	11	.557	90	19	12
.524	85	11	17	.558	91	2	19
.525	85	15	0	.559	91	6	1
.526	85	18	6	.560	91	9	8
.527	86	1	12	.561	91	12	14
.528	86	4	19	.562	91	15	20
.529	86	8	1	.563	91	19	3
.530	86	11	8	.564	92	2	9
.531	86	14	14	.565	92	5	16
.532	86	17	20	.566	92	8	22
.533	87	1	3	.567	92	12	4
.534	87	4	9	.568	92	15	11
.535	87	7	16	.569	92	18	17
.536	87	10	22	.570	93	2	0
.537	87	14	4	.571	93	5	6
.538	87	17	11	.572	93	8	12
.539	88	0	17	.573	93	11	19
.540	88	4	0	.574	93	15	1
.541	88	7	6	.575	93	18	8
.542	88	10	12	.576	94	1	14
.543	88	13	19	.577	94	4	20
.544	88	17	1	.578	94	8	3
.545	89	0	8	.579	94	11	9
.546	89	3	14	.580	94	14	16

If 200 grains of ore give: FINE METAL.	One ton of ore will yield: FINE METAL.			If 200 grains of ore give: FINE METAL.	One ton of ore will yield: FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.581	94	17	22	.615	100	9	0
.582	95	1	4	.616	100	12	6
.583	95	4	11	.617	100	15	12
.584	95	7	17	.618	100	18	19
.585	95	11	0	.619	101	2	1
.586	95	14	6	.620	101	5	8
.587	95	17	12	.621	101	8	14
.588	96	0	19	.622	101	11	20
.589	96	4	1	.623	101	15	3
.590	96	7	8	.624	101	18	9
.591	96	10	14	.625	102	1	16
.592	96	13	20	.626	102	4	22
.593	96	17	3	.627	102	8	4
.594	97	0	9	.628	102	11	11
.595	97	3	16	.629	102	14	17
.596	97	6	22	.630	102	18	0
.597	97	10	4	.631	103	1	6
.598	97	13	11	.632	103	4	12
.599	97	16	17	.633	103	7	19
.600	98	0	0	.634	103	11	1
.601	98	3	6	.635	103	14	8
.602	98	6	12	.636	103	17	14
.603	98	9	19	.637	104	0	20
.604	98	13	1	.638	104	4	3
.605	98	16	8	.639	104	7	9
.606	98	19	14	.640	104	10	16
.607	99	2	20	.641	104	13	22
.608	99	6	3	.642	104	17	4
.609	99	9	9	.643	105	0	11
.610	99	12	16	.644	105	3	17
.611	99	15	22	.545	105	7	0
.612	99	19	4	.546	105	10	6
.613	100	2	11	.647	105	13	12
.614	100	5	17	.648	105	16	19

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.649	106	0	1
.650	106	3	8
.651	106	6	14
.652	106	9	20
.653	106	13	3
.654	106	16	9
.655	106	19	16
.656	107	2	22
.657	107	6	4
.658	107	9	11
.659	107	12	17
.660	107	16	0
.661	107	19	6
.662	108	2	12
.663	108	5	19
.664	108	9	1
.665	108	12	8
.665	108	15	14
.667	108	18	20
.668	109	2	3
.669	109	5	9
.670	109	8	16
.671	109	11	22
.672	109	15	4
.673	109	18	11
.674	110	1	17
.675	110	5	0
.676	110	8	6
.677	110	11	12
.678	110	14	19
.679	110	18	1
.680	111	1	8
.681	111	4	14
.682	111	7	20

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.683	111	11	3
.684	111	14	9
.685	111	17	6
.686	112	0	22
.687	112	4	4
.688	112	7	11
.689	112	10	17
.690	112	14	0
.691	112	17	6
.692	113	0	12
.693	113	3	19
.694	113	7	1
.695	113	10	8
.696	113	13	14
.697	113	16	20
.698	114	0	3
.699	114	3	9
.700	114	6	16
.701	114	9	22
.702	114	13	4
.703	114	16	12
.704	114	19	17
.705	115	3	0
.706	115	6	6
.707	115	9	12
.708	115	12	19
.909	115	16	1
.710	115	19	8
.711	116	2	14
.712	116	5	20
.713	116	9	3
.714	116	12	9
.715	116	15	16
.716	116	18	22

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.717	117	2	4	.751	122	13	16
.718	117	5	11	.752	122	16	12
.719	117	8	17	.753	122	19	19
.720	117	12	0	.754	123	3	1
.721	117	15	6	.755	123	6	8
.722	117	18	12	.756	123	9	14
.723	118	1	19	.757	123	12	20
.724	118	5	1	.758	123	16	3
.725	118	8	8	.759	123	19	9
.726	118	11	14	.760	124	2	16
.727	118	14	20	.761	124	5	22
.728	118	18	3	.762	124	9	4
.729	119	1	9	.763	124	12	11
.730	119	4	16	.764	124	15	17
.731	119	7	22	.765	124	19	0
.732	119	11	4	.766	125	2	6
.733	119	14	11	.767	125	5	12
.734	119	17	17	.768	125	8	19
.735	120	1	0	.769	125	12	1
.736	120	4	6	.770	125	15	8
.737	120	7	12	.771	125	18	14
.738	120	10	19	.772	126	1	20
.739	120	14	1	.773	126	5	3
.740	120	17	2	.774	126	8	9
.741	121	0	14	.775	126	11	16
.742	121	3	20	.776	126	14	22
.743	121	7	3	.777	126	18	4
.744	121	10	9	.778	127	1	11
.745	121	13	6	.779	127	4	17
.746	121	16	22	.780	127	8	0
.747	122	0	4	.781	127	11	6
.748	122	3	11	.782	127	14	12
.749	122	6	17	.783	127	17	19
.750	122	10	0	.784	128	1	1

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.785	128	4	8	.819	133	15	9
.786	128	7	14	.820	133	18	16
.787	128	10	20	.821	134	1	22
.788	128	14	3	.822	134	5	4
.789	128	17	9	.823	134	8	11
.790	129	0	16	.824	134	11	17
.791	129	3	22	.825	134	15	0
.792	129	7	4	.826	134	18	6
.793	129	10	11	.827	135	1	12
.794	129	13	17	.828	135	4	19
.795	129	17	0	.829	135	8	1
.796	130	0	6	.830	135	11	8
.797	130	3	12	.831	135	14	14
.798	130	6	19	.832	135	17	20
.799	130	10	1	.833	136	1	3
.800	130	13	8	.834	136	4	9
.801	130	16	14	.835	136	7	16
.802	130	19	20	.836	136	10	22
.803	131	3	3	.837	136	14	4
.804	131	6	9	.838	136	17	11
.805	131	9	16	.839	137	0	17
.806	131	12	22	.840	137	4	0
.807	131	16	4	.841	137	7	6
.808	131	19	11	.842	137	10	12
.809	132	2	17	.843	137	13	19
.810	132	6	0	.844	137	17	1
.811	132	9	6	.845	138	0	8
.812	132	12	12	.846	138	3	14
.813	132	15	19	.847	138	6	20
.814	132	19	1	.848	138	10	3
.815	133	2	8	.849	138	13	19
.816	133	5	14	.850	138	16	16
.817	133	8	20	.851	138	19	22
.818	133	12	3	.852	139	3	4

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.853	139	6	11	.887	144	17	12
.854	139	9	17	.888	145	0	19
.855	139	13	0	.889	145	4	1
.856	139	16	6	.890	145	7	8
.857	139	19	12	.891	145	10	14
.858	140	2	19	.892	145	13	20
.859	140	6	1	.893	145	17	3
.860	140	9	8	.894	146	0	9
.861	140	12	14	.895	146	3	16
.862	140	15	20	.896	146	6	22
.863	140	19	3	.897	146	10	4
.864	141	2	9	.898	146	13	11
.865	141	5	16	.899	146	16	17
.866	141	8	22	.900	147	0	0
.867	141	12	4	.901	147	3	6
.868	141	15	11	.902	147	6	12
.869	141	18	17	.903	147	9	19
.870	142	2	0	.904	147	13	1
.871	142	5	6	.905	147	16	8
.872	142	8	12	.906	147	19	14
.873	142	11	19	.907	148	2	2
.874	142	15	1	.908	148	6	3
.875	142	18	8	.909	148	9	9
.876	143	1	14	.910	148	12	16
.877	143	4	20	.911	148	15	21
.878	143	8	3	.912	148	19	4
.879	143	11	9	.913	149	2	11
.880	143	14	16	.914	149	5	17
.881	143	17	22	.915	149	9	0
.882	144	1	4	.916	149	12	6
.883	144	4	11	.917	149	15	12
.884	144	7	17	.918	149	18	19
.885	144	11	0	.919	150	2	1
.886	144	14	6	.920	150	5	8

If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.
.921	150	8	14
.922	150	11	20
.923	150	15	3
.924	150	18	9
.925	151	1	16
.926	151	4	22
.927	151	8	4
.928	151	11	11
.929	151	14	17
.930	151	18	0
.931	152	1	6
.932	152	4	12
.933	152	7	19
.934	152	11	1
.935	152	14	8
.936	152	17	14
.937	153	0	20
.938	153	4	3
.939	153	7	9
.940	153	10	16
.941	153	13	22
.942	153	17	4
.943	154	0	11
.944	154	3	17
.945	154	7	0
.946	154	10	6
.947	154	13	12
.948	154	16	19
.949	155	0	1
.950	155	3	8
.951	155	6	14
.952	155	9	20
.953	155	13	3
.954	155	16	9
.955	155	19	16
.956	156	2	22
.957	156	6	4
.958	156	9	11
.959	156	12	17
.960	156	16	0
.961	156	19	6
.962	157	2	12
.963	157	5	19
.964	157	9	1
.965	157	12	8
.966	157	15	14
.967	157	18	20
.968	158	2	3
.969	158	5	9
.970	158	8	16
.971	158	11	22
.972	158	15	4
.973	158	18	11
.974	159	1	17
.975	159	5	0
.976	159	8	6
.977	159	11	12
.978	159	14	19
.979	159	18	1
.980	160	1	8
.981	160	4	14
.982	160	7	20
.983	160	10	3
.984	160	14	9
.985	160	17	6
.986	161	0	22
.987	161	4	4
.988	161	7	11

If 200 grains of ore give:	One ton of ore will yield:			If 200 grains of ore give:	One ton of ore will yield:		
FINE METAL.	FINE METAL.			FINE METAL.	FINE METAL.		
gr.	oz.	dwts.	grs.	gr.	oz.	dwts.	grs.
.989	161	10	17	.995	162	10	8
.990	161	14	0	.996	162	13	14
.991	161	17	6	.997	162	16	20
.992	162	0	12	.998	163	0	3
.993	162	3	19	.999	163	3	9
.994	162	7	1	1.000	163	6	16

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